INTERNATIONAL CENTRE FOR THE STUDY OF THE PRESERVATION AND THE RESTORATION OF CULTURAL PROPERTY
CENTRE INTERNATIONAL D'ETUDES POUR LA CONSERVATION ET LA RESTAURATION DES BIENS CULTURELS

ICCROM

Mortars, Cements and Grouts used in the Conservation of Historic Buildings

Mortiers, ciments et coulis utilisés dans la conservation des bâtiments historiques

Symposium 3-6.11.1981 Rome

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INTRODUCTION

The problem of the use (and abuse) of mortars in historic buildings must be considered a prominent one, if one thinks of the vital role that mortar properties play in the funcioning of a structure and its outer skin.

Yet this domain has received, up to the present moment, far less attention than that of the conservation of stone, a material which is comparatively less important from a quantitative point of view but is more in the public eye because many important works of art or famous structures are carved in stone.

This lack of attention leads to a series of unfortunate consequences such as those given below :

a. mortars and renderings are an important source of historical information, which is insufficiently exploited ; their study is neglected, and original materials are often destroyed without preservation of samples and proper documentation ;

b. injection of grouts is used to stabilize old masonry without any theoretical consideration of requirements for the material to be injected and of the future behaviour of the grouted structures under environmental stresses ;

c. mortars of insufficient porosity and excessive strength, which form dangerous by-products in their setting reactions. are frequently used in contact with old masonry and even near important works of art (mural paintings, stuccoes or bas-reliefs).

This list is by no means exhaustive ; undoubtedly other lamentable effects of the neglect of mortar studies can be easily added.

With such considerations in mind, ICCROM decided to devote its 1981 annual technical symposium to mortars and grouts in old buildings, including both the study of old materials and the use of new ones.

A survey of the available technical literature allowed us to draft a list of persons active in the field, who could make an interesting contribution to the symposium. We are quite conscious, however, that some relevant research programmes and interested specialists may have never been aware of the meeting, as it was publicized only in journals and newsletters devoted to conservation, and our initial list of invited experts was obviously incomplete.

Notwithstanding such limitations, we were fortunate enough to bring together about seventy professionals who attended three days (3, 4 and 5 November, 1981) of meetings devoted to the presentation and discussion of 26 papers.

ICCROM also presented two symposium papers produced in its research programmes. The studies were carried out by former course participants under the supervision of ICCROM technical staff and with the cooperation of scientific laboratories in Rome.

It is a tradition of ICCROM technical symposia that an attempt is made to give some propulsion to further activities in the domain under consideration. To that purpose, one half of the last day was devoted to the informal meetings of two working groups : one on the study of ancient mortars, the other on the use of mortars and grouts in conservation.

The two groups prepared recommendations which indicate the domain and objectives of possible future research projects considered of importance for the study and the conservation of ancient buildings.

These recommendations were approved in a short final session of the symposium and are included (in both French and English) at the end of this volume.

On 6 November 1981 the symposium was closed by an excursion to Tuscania, about 60 miles north of Rome, which allowed the participants to review the use and abuse of mortars in the conservation of an earthquake-stricken medieval town.

In the publication of this volume, priority was given to speed and timeliness and, therefore, to simplicity. Papers were reproduced as presented by the authors, with little or no editing.

Authors are responsible for the choice and the presentation of the facts contained in their signed articles and for the opinions expressed therein, which are not necessarily those of ICCROM.

Discussions could not be included, but an echo of them is reflected in the recommendations drafted by the working groups at the end of the meeting.

We hope that the publication of the symposium proceedings will help further expansion of mortar studies, particularly along the lines which now appear of prominent historical and technological interest, in particular :

1. history of the technology of ancient mortars, based on large numbers of accurate, standardized analyses ;

2. development of a system of standard tests, and specifications explicitly meant for conservation mortars ;

3. study of new compositions of mortars designed to fulfil specific conservation needs.

INTRODUCTION

Le problème de l'utilisation (et de l'abus) du mortier dans les monuments historiques doit être considéré comme majeur si l'on pense au rôle essentiel joué par le mortier dans le fonctionnement de la structure et sa couche extérieure.

Ce domaine a pourtant reçu moins d'attention que celui de la conservation de la pierre, un matériau comparativement moins important mais davantage en vue, beaucoup d'oeuvres d'art ou de sculptures célèbres étant taillées dans la pierre.

Ce manque d'attention a amené toute une série de conséquences indésirables telles que celles indiquées ci-dessous :

a. les mortiers et les enduits sont une source importante d'informations historiques, non suffisamment exploitée. Leur étude est négligée et des matériaux originaux sont souvent détruits sans qu'on ait effectué des prélèvements d'échantillons et sans documentation adéquate.

b. l'injection des liants est utilisée pour stabiliser des maçonneries anciennes sans aucune considération théorique des qualités requises pour l'injection et du comportement ultérieur des structures ainsi injectées sous les pressions de l'environnement.

c. des mortiers d'une porosité insuffisante et d'une résistance trop grande ou qui réagissent par la formation de résidus dangereux au cours de leur phase de prise, sont souvent mis en contact avec des maçonneries anciennes ou même utilisés à proximité d'oeuvres d'art de grande valeur (peintures murales, stucs, bas-reliefs).

Cette liste est incomplète : on pourrait facilement trouver d'autres exemples de ces conséquences désastreuses dues au manque d'étude sur les mortiers.

Ayant à l'esprit les considérations énoncées, l'ICCROM a décidé de consacrer son colloque technique annuel de 1981 aux mortiers et liants dans les bâtiments anciens, comprenant à la fois l'étude des vieux matériaux et l'utilisation des nouveaux.

L'examen de la documentation technique à disposition nous a permis de dresser une liste de personnes actives dans le domaine et susceptibles d'apporter une contribution intéressante au colloque. Nous sommes cependant conscients du fait que certains programmes de recherche analogues ainsi que des spécialistes intéressés ont pu ne pas avoir connaissance de cette réunion, laquelle n'a été annoncée que dans les journaux et chroniques consacrés à la conservation. De plus notre liste initiale d'experts invités était évidemment incomplète.

En dépit de ces limitations nous avons heureusement pu rassembler environ 70 professionnels qui, pendant trois jours (les 3, 4 et 5 novembre), ont assisté à des réunions consacrées à la présentation et à la discussion de 26 exposés.

ICCROM a également présenté deux rapports au colloque, élaborés par ses programmes de recherche. Les études avaient été entreprises par d'anciens participants aux cours sous la conduite du personnel technique de l'ICCROM et avec la collaboration de laboratoires scientifiques de Rome.

Il est de tradition pour les colloques techniques de l'ICCROM d'essayer de favoriser l'essort des activités du domaine à l'étude. A cette fin, deux groupes de travail se sont rencontrés la moitié du dernier jour pour étudier : l'un, les mortiers anciens et l'autre, l'utilisation des mortiers et liants dans la conservation.

Les deux groupes ont préparé des recommandations indiquant le champ et les objectifs pour des projets de recherche ultérieurs possibles considérés importants pour l'étude et la conservation des bâtiments anciens.

Ces recommandations ont été approuvées lors d'une brève session finale du colloque et sont comprises à la fin de ce volume, en anglais et en français.

Le colloque s'est achevé le 6 novembre par une excursion à Tuscania, à environ 100 km au nord de Rome, qui a permis aux participants de noter l'utilisation et l'abus du mortier dans la conservation d'une ville médiévale endommagée par un tremblement de terre

Pour la publication de ce volume, les priorités ont été : rapidité et à propos, et par conséquent simplicité. Les exposés ont été imprimés tels que présentés par les auteurs avec très peu ou aucune correction.

Les auteurs sont responsables du choix et de la présentation des faits contenus dans leurs articles et les opinions exprimées ne reflètent pas nécessairement celles de l'ICCROM.

Les discussions n'ont pu être inclues mais on en trouvera un écho dans les recommandations esquissées par les groupes de travail à la fin de la réunion.

Nous espérons que la publication du déroulement du colloque aidera à promouvoir une étude sur les mortiers en mettant l'accent sur des points qui semblent d'un intérêt historique et technologique particulier, par exemple :

1. Histoire de la technologie des mortiers anciens, basée sur des analyses précises et standardisées.

2. Développement d'un système standard d'essais et de spécifications réservées aux mortiers de conservation.

3. Etude de nouveaux mélanges de mortiers destinés à satisfaire des besoins spécifiques en conservation.

Section 1 :

Mortars for renderings, repointing and masonry repairs.

Mortiers pour enduits, rejointement et réparation des maçonneries.

EXPERIENCES PRATIQUES

AVEC DES CREPIS A BASE DE CHAUX

Furlan Vinicio*

SUMMARY

This contribution deals with experiences using lime paste mortars for wall plastering. The basic conditions for the realisation are defined and the advantages and disadvantages of the hydraulic binders are discussed.

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INTRODUCTION

La réfection des crépis des bâtiments anciens pose actuellement de sérieux problèmes. Bien que voué par sa fonction à se "sacrifier", et donc à être refait tôt ou tard, le crépi, au-delà de son rôle de protection, représente une valeur esthétique étroitement liée à l'architecture, donc aux formes et aux modes de construction. La nature même des matériaux, impliquant une mise en oeuvre particulière, contribue à créer une image personnalisée du crépi.

Pour les bâtiments dont la construction est antérieure à la deuxième moitié du XIXe siècle, seuls les crépis à la chaux semblent satisfaire pleinement aux exigences des conservateurs soucieux de respecter et sauvegarder cette image. Par ailleurs, le crépi à la chaux se justifie aussi au point de vue technologique. Lors des restaurations, les réfections de crépis à la chaux sont cependant très rares, particulièrement en raison du manque de main-d'oeuvre qualifiée. Le maçon moderne a tendance à considérer la pratique du mortier à la chaux comme une technique démodée incompatible avec les méthodes de travail modernes; de ce fait, il est animé par un sentiment de peur face à des procédés dont il a depuis longtemps perdu la maîtrise; il craint les complications et les risques de dommages puisque le mortier à la chaux a un durcissement très lent et une faible résistance mécanique. Si on ne domine pas la technique et qu'on travaille sans précaution, il y a en effet un risque élevé de dégâts, notamment par le gel. Dans ce cas, le recours à des mortiers aux liants hydrauliques paraît justifié. Pourtant, le crépi à la chaux a fait ses preuves. Il n'est pas rare en effet de voir, même dans les pays humides et froids, des crépis à la chaux ayant dépassé sans dommage 100 à 200 ans.

Développés à partir de la deuxième moitié du XIXe siècle (1, 2) suite à la généralisation de l'usage du ciment portland, les crépis à base de liants hydrauliques ne sont guère appréciés par les conservateurs. Il faut admettre que ce genre de crépis a causé beaucoup de laideur et de dommages qui expliquent l'aversion qui se manifeste aujourd'hui pour le ciment dans le milieu de la conservation. Nous pensons toutefois que c'est surtout l'emploi abusif du ciment qui en est responsable. Quant aux enduits modernes, à base de résines synthétiques, admettons tout simplement qu'ils sont inacceptables pour des bâtiments anciens ayant une valeur historique et architecturale. Dans ce contexte, consultés souvent par les services des monuments historiques, nous avons essayé, non sans peine, de promouvoir le retour aux crépis à la chaux. Des contacts personnels et des cours (3) pour sensibiliser les praticiens au problème ont permis la réalisation d'un certain nombre d'expériences pratiques. Grâce à la collaboration enthousiaste de praticiens compétents, ces expériences nous ont permis de mieux connaître la technologie du crépi à la chaux, d'en saisir les particularités et les limites et d'apprécier les avantages pouvant découler de l'utilisation, en quantité modérée, de liants hydrauliques. Ce sont les résultats de ces expériences que nous résumerons ici. Fruit de la collaboration avec des praticiens, notre contribution a un caractère et un but éminemment pratique.

ROLE ET PROPRIETES REQUISES DES CREPIS

Il est utile de rappeler que les exigences auxquelles doit satisfaire un crépi sont nombreuses et complexes. Les exigences que l'on peut considérer comme essentielles sont les suivantes :

- *bonne adhérence* au support pour éviter tout décollement
- *déformabilité* permettant de supporter les déformations du support sans fissuration ni décollement
- *étanchéité* à l'eau de pluie et bonne tenue en général aux intempéries (bonne durabilité)
- *perméabilité* à la vapeur d'eau venant de l'intérieur du bâtiment et à l'eau qui aurait pénétré dans la maçonnerie par des fissures
- *aspect satisfaisant*, durable : couleur et texture correspondant aux effets désirés

Pour les maçonneries modernes, les crépis à base de liants hydrauliques et de chaux exécutés en trois couches, dont la composition est adaptée à la nature des matériaux, satisfont aux exigences énumérées. Laideur éventuelle et dégradation prématurée doivent être attribuées essentiellement à une mauvaise conception ou à une exécution défaillante (4). Dans le cas des bâtiments anciens ayant une valeur historique et architecturale, aux exigences formulées on doit ajouter le souci d'une certaine "réversibilité" de l'intervention. Nous entendons par là la possibilité de décrépir les bâtiments, dans un avenir plus ou moins lointain, sans endommager la maçonnerie; ceci afin de préserver l'intégrité du monument et à en faciliter la lecture aux chercheurs des générations futures.
Au premier abord, il paraît difficile de concilier deux concepts aussi opposés que durabilité et réversibilité. Pourtant, les mortiers à la chaux satisfont à la définition de réversibilité donnée ci-dessus.
Le vieillissement et la dégradation naturelle des crépis à la chaux, contrairement à ce qui a été constaté bien souvent avec des crépis au ciment, n'entraîne pratiquement aucun dommage pour la maçonnerie. Ceci est essentiellement dû au fait que le mortier à la chaux a une résistance mécanique toujours inférieure à celle du support.
En adoptant ce principe, il est à notre avis possible, dans certaines situations, de profiter des avantages indéniables des liants hydrauliques (rapidité de durcissement, par exemple) tout en évitant les inconvénients. Cela présuppose une réduction drastique des dosages

en liant communément pratiqués. La réduction du dosage ainsi que le choix judicieux de la composition permet d'obtenir, non seulement au point de vue technologique mais également esthétique, des résultats satisfaisants. En fonction des caractéristiques et de la valeur intrinsèque des bâtiments et des contraintes diverses qui, directement ou indirectement, peuvent avoir une incidence déterminante sur la qualité et la durabilité des travaux envisagés, nous avons expérimenté grosso-modo trois types de crépis à base de :

a) chaux uniquement
b) chaux additionnée de quantités modérées de liants hydrauliques
c) chaux et liants hydrauliques

CREPIS A LA CHAUX

La réalisation d'un crépi avec des mortiers liés uniquement à la chaux hydratée est à notre avis envisageable sans crainte si les conditions suivantes sont réunies :

- présence d'avant-toits protégeant les façades fortement exposées à la pluie
- suppression de toute remontée capillaire et des défauts de construction pouvant entraîner l'accumulation et la stagnation d'eau dans le crépi
- disponibilité de main-d'oeuvre qualifiée connaissant la technologie du mortier à la chaux ou apte à l'assimiler
- garantie que les travaux auront lieu selon un rythme bien défini et dans la période préétablie (début des travaux lorsque tout risque de gel est écarté et fin des travaux bien avant la période de gel)

Il est extrêmement important que le mortier à la chaux puisse "durcir" avant la période hivernale car, à l'état non carbonaté, il manifeste une hydrophilie marquée qui augmente considérablement les risques de destruction par le gel. Après carbonatation, il se mouille moins facilement et restitue rapidement l'eau absorbée. En outre, il est très perméable à la vapeur d'eau et peu sensible aux variations de l'humidité relative de l'air. Par contre, en raison de sa faible résistance mécanique, il demeure toujours sensible à certains facteurs de dégradation tels que :

a) les chocs mécaniques
b) la cristallisation de sels
c) le gel, s'il y a possibilité de saturation en eau (pieds de murs soumis à des remontées capillaires, par exemple)

On sait que la résistance au gel d'un mortier à la chaux saturé d'eau est pratiquement nulle.

Composition des mortiers

Selon les auteurs anciens, et Vitruve (5) en particulier, qui jusqu'à la première moitié du XIXe siècle constitue la principale source de renseignements sur les mortiers de chaux (6), le rapport volumétrique entre granulat et chaux en pâte peut varier de 2-3 à 1. L'examen de nombreux échantillons d'anciens crépis a montré que la quantité de sable et de chaux varie dans une telle proportion. Ayant constaté que les meilleurs mortiers sont en général relativement "maigres", nous avons adopté le dosage de chaux en pâte qui correspond au remplissage complet des vides du sable. Des essais de laboratoire, dont la réalisation est assez délicate, semble prouver qu'il s'agit de la composition optimale qui, au point de vue de la résistance mécanique, donne les meilleurs résultats. En pratique, selon le type et la granulométrie du sable le rapport en question peut varier grosso-modo de 2,5-3 à 1. Pour un sable de rivière (Rhône 0/3, par exemple), le rapport entre volume des pleins et volume des vides est d'environ 2,7 à 1. Le tableau I donne un exemple de formulation type de crépi à la chaux réalisé en trois couches. Dans le calcul des volumes il est tenu compte du foisonnement du sable, car sur les chantiers les granulats sont pratiquement toujours humides et on a tendance à l'oublier. Connaissant l'indice des vides et la teneur en eau du sable, le dosage peut être calculé évidemment avec précision. A notre avis il est surtout important d'éviter les erreurs de dosage grossières.

Granulats / Liants	Rempochage	1ère couche ou couche d'accrochage		2ème couche ou couche de fond		3ème couche ou couche de finition	
	parties en volume	parties en volume	épais. mm	parties en volume	épais. mm	parties en volume	épais. mm
sable 0/8	11	-		9		-	
sable 0/3	-	8	3-5	-	10-20	10	3-5
chaux en pâte	3	3		3		3	

Tableau I : Exemple de composition de crépi à la chaux en 3 couches.

La plupart de nos expériences ont été réalisées avec une bonne chaux en pâte ayant un rendement (volume de pâte en litres obtenu par extinction de 1 kg de CaO) d'environ 2,7 et ayant séjourné en fosse environ 6 mois. Il est à souligner que dans le cas d'un mortier fabriqué avec de la chaux en pâte, le dosage en liant effectif ($ca(OH)_2$), calculé par m^3 de mortier, est étonnamment faible; pour un mortier "plein" il est inférieur à 200 kg/m^3. Le dosage est d'autant plus faible que la chaux est de bonne qualité (rendement élevé).

Au point de vue de la maniabilité du mortier, les sables de gravière se sont révélés les meilleurs. Ils ont généralement une meilleure granulométrie que les sables de rivière et, à dosage égal en chaux, donnent des mortiers plus plastiques, plus faciles à mettre en oeuvre. La fabrication du mortier, selon la méthode traditionnelle, dans une grande caisse à l'aide de pelle, rateau et pelle-bêche, est un procédé long et fastidieux (pour fabriquer 100 litres de mortier, deux personnes employent au moins 15-20 minutes). Au moyen d'un malaxeur horizontal on peut obtenir de très bons résultats avec économie de temps et de main-d'oeuvre (durée de malaxage de 5 à 10 minutes). Prêt à l'emploi, le mortier doit avoir une consistance plastique. Normalement, avec un sable moyennement humide et une bonne chaux, on obtient la consistance voulue sans ou avec une faible adjonction d'eau. Tout excès d'eau doit être évité.

Crépissage

De manière générale, lorsqu'on crépit avec des mortiers à la chaux (ou chaux avec une faible addition de liants hydrauliques), afin de favoriser la carbonatation, il convient d'espacer le plus possible les couches en tenant compte entre outre de leur épaisseur. Si la couche de fond est très épaisse, l'application doit être faite en deux mains. En fonction des conditions météorologiques, le délai d'attente entre une couche et la suivante peut varier de 3 à 6 semaines.
Le "séchage" est indispensable pour permettre la carbonatation, mais la dessiccation ne doit pas être trop brutale. Au cours de la première semaine, le mortier doit être maintenu humide afin d'éviter la formation de grandes fentes de retrait. D'autre part, on a pu remarquer que le séchage brutal a aussi une incidence néfaste sur la résistance mécanique finale du mortier. Contrairement à l'opinion couramment émise, qu'il faut abondamment mouiller la maçonnerie avant le début des travaux de crépissage, il est conseillé de l'humecter avec modération en fonction de la nature et de la capacité d'absorption des matériaux, et d'entretenir ensuite l'humidité des couches fraîchement appliquées pendant la première semaine, et cela en fonction aussi des conditions de séchage.

Suivant l'importance des recharges à effectuer, le mortier des rempochages, après projection et séchage partiel (absorption d'eau par le support et perte par évaporation), est "serré" à la main ou à la truelle ou avec autre outil adéquat. Le mortier de la couche d'accrochage est simplement projeté énergiquement. La couche de fond est également projetée avec force et "tirée" à la truelle pour l'égaliser (on ne pratique normalement pas d'autres formes de "serrage"). La technique d'application de la couche de finition dépend du rendu que l'on veut obtenir. Dans le cas d'un *crépi lissé à la truelle* par exemple, le mortier de la couche de finition est projeté et lissé une première fois avec le dos de la truelle. Après une période d'attente variant de 15 à 60 minutes, pour permettre l'élimination partielle de l'eau de gâchage, on effectue un deuxième et ultime lissage.

Remarque : En principe, le "serrage" par frottement, après élimination d'une partie de l'eau de gâchage, permet d'éliminer les éventuelles fissures de retrait et d'obtenir, en augmentant la compacité du mortier, une meilleure résistance mécanique. Le retrait du mortier à la chaux, lié au départ de l'eau, a lieu très rapidement.

S'il s'agit d'un *crépi tiré à la truelle*, le mortier de la couche de finition est "tiré" une première fois après projection, puis une deuxième fois après 5 à 10 minutes d'attente.

D'autres types de finition sont envisageables en fonction de l'architecture ou d'après l'analyse de témoins d'anciens crépis. La coloration éventuelle du crépi à la fresque ne pose pas de problèmes particuliers. A notre avis il faut éviter l'application sur le mortier frais de badigeons épais qui formant une pellicule risquent de trop retarder la carbonatation.

Note : la couche de finition des crépis anciens est parfois teintée dans la masse avec des pigments divers ou du tuileau. Le remplacement d'une partie du granulat avec du tuileau, pratique très ancienne utilisée notamment pour les enduits recouvrant les réservoirs et les conduites d'eau, permet d'obtenir des mortiers d'excellente qualité.

CREPIS A LA CHAUX AVEC ADDITION MODEREE DE LIANTS HYDRAULIQUES

L'addition d'une quantité modérée de liants hydrauliques (ciment portland ou ciment portland blanc, par exemple) à un mortier de chaux n'est pas, à notre avis, préjudiciable. Un tel mortier garde fondamentalement les propriétés du mortier à la chaux (perméabilité à la vapeur, par exemple) tout en ayant une meilleure résistance mécanique. Même au point de vue esthétique, pouvant être "travaillé" comme le mortier à la chaux, il permet d'obtenir des résultats satisfaisants. L'expérience nous a montré que ce genre de mortier remplace avantageusement le mortier à la chaux lorsque le crépi est particulièrement exposé aux sollicitations mécaniques (chocs) et aux intempéries (façades non protégées par avant-toits, soumises au ruissellement, contreforts, murs avec couronnement sans protection, etc.). En particulier, on peut éviter les dégâts dus à la stagnation d'eau aux pieds des façades. Le mortier à la chaux, demeurant humide, ne durcit pas et a tendance à s'effriter sous l'effet du gel.

Dans la formulation des mortiers (voir tableau I), une partie en volume de chaux en pâte est remplacée par le volume équivalent (volume apparent) de ciment (le dosage en ciment du mortier est donc d'environ 100 kg/m^3).

Remarque : Pour éviter des éventuels dégâts dus à la solubilisation et à la cristallisation de sels, il est judicieux de choisir les ciments les plus pauvres en alcalis.

CREPIS A BASE DE LIANTS HYDRAULIQUES

Les principaux avantages des crépis à base de liants hydrauliques sont d'être familiers aux maçons modernes et de pouvoir être utilisés dans des conditions défavorables (à l'approche de l'hiver, par exemple) sans trop de risques de malfaçons ou de dégâts prématurés par le gel. La laideur et les dommages causés à la longue aux maçonneries (arrachements sous l'effet des contraintes dues aux variations hygrométriques ou thermiques, par exemple) qu'on reproche à ce genre de crépis ne sont à notre avis que partiellement justifiés. L'examen de ces cas montre en effet que laideur et dégâts sont surtout imputables à des dosages en liants beaucoup trop élevés et en particulier à l'emploi abusif de ciment qui donne des crépis trop durs et peu perméables à la vapeur d'eau.

C'est la raison qui nous a amenés à développer et à expérimenter avec succès depuis plusieurs années des mortiers pour crépis relativement peu dosés en liants et surtout en ciment. Le tableau II donne un exemple de ce type de formulation.

Granulats / Liants	Rempochage	1ère couche ou couche d'accrochage		2ème couche ou couche de fond		3ème couche ou couche de finition	
	parties en volume	parties en volume	épais. mm	parties en volume	épais. mm	parties en volume	épais. mm
sable 0-8	12	-		12		-	
sable 0/3	-	12		-		12	
c.p. normal	1/2	1		1/2		-	
c.p. blanc	-	-	3-5	-	12-15	1	3-5
chaux hydraulique	1 1/2	2 1/2		2		-	
chaux hydratée (poudre)	-	-		1		3	
chaux hydratée (pâte)	1	-		-		-	

Tableau II : Exemple de formulation pour crépis à base de liants hydrauliques.

Dans le calcul il a été tenu compte d'un foisonnement moyen du sable. Le dosage global en liants de chaque couche est approximativement de 300 kg/m^3 avec une proportion en liants "forts" décroissant de la première couche à la troisième. La couche de finition contenant beaucoup de chaux hydratée en poudre (à remplacer éventuellement par de la chaux en pâte) permet d'obtenir sur le plan esthétique une structure comparable à celle du mortier de chaux.

CONCLUSIONS

L'expérience nous a montré que :

- un crépi à la chaux peut être réalisé avec succès lorsque plusieurs conditions favorables sont réunies
- l'utilisation en quantité modérée de liants hydrauliques permet, dans certaines situations, de conférer au mortier à la chaux une durabilité accrue
- dans des conditions défavorables, on peut crépir sans trop d'inconvénients avec des mortiers à base de liants hydrauliques, à condition d'adopter des formulations conformes aux exigences particulières de la restauration.

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MORTARS, CEMENTS AND GROUTS FOR CONSERVATION AND REPAIR.
SOME URGENT NEEDS FOR RESEARCH.

INGMAR HOLMSTRÖM*

SUMMARY

In the Scandinavian building trade, lime has long been replaced by portland cement, but cement has proved to cause damage to the original fabric because it is too hard, inflexible, and impermeable. The re-use of lime has often failed, as the desired strength is not obtained.

Investigating old mortars demands real teamwork, with historians, technicians, scientists and masons working hand in hand.

We still cannot study and compare such factors as strength or permeability in old and new mortars because we have no proper testing methods. Nor do we have proper methods for taking old, or making new samples, or testing such samples. Existing methods are designed for new portland cement mortars.

When better methods are developed, we should be able to reconstruct mortars and also construct entirely new compositions.

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GENERAL EXPERIENCES

To find the right kind of mortar or grout for the conservation of historic buildings is one of the most difficult technical restoration problems we have in Scandinavia. The original mortars were based on slaked lime, a binder out of use in the building trade for over a generation. The lime has been replaced by portland cement and by synthetic resins or mixtures with these binders as primary ingredients. This has provided some advantages but also some disadvantages. Unfortunately, we have found that the damage caused by the new materials is more serious than the advantages they bring. I have also found that such problems are more or less the same all over the world.

Replacing lime with portland cement gives a much quicker and safer hardening and binding and at the same time a stronger material that is less permeable to both liquids and gases.

Replacing lime with synthetic resins gives a much safer binding, better adhesion, a good strength, and better resistance to weathering, especially in polluted environments.

The problem, however, is that these positive properties often turn negative, or cause unforseen negative side effects, and the result is a more or less damaged building.

From the technical point of view we know that lime mortars (as well as lime wash) have suitable properties - if they are of a good quality and well done. Unfortunately, this is too often not the case. The hardening process - the carbonization - is poor or of uneven strength. The lime mortar must be mixed and treated correctly, and the climatic conditions must be rather precise during the hardening process, a process that lasts for weeks. If it does not work, the climate is either too cold, too wet, or too dry. In our climate the failures are mostly due to low temperatures and high moisture.

It is very easy to understand why cement has increasingly taken over, as it has a more reliable hardening process. Cement always works safely if the temperature in the material is not below zero and there is at least some moisture present. These characteristics have become more and more necessary as the erection of a building has turned from summer season to year-round work during this century.

In the case of historic buildings, we have noticed more and more disadvantages with mortars and grouts containing portland cement. They are too hard and dense with not enough flexibility and permeability. They also contain soluble salts.

Swedish practice

We have found that mortars and grouts used for repair must be equal to the original to cooperate in the right way: equal in strength, adhesion, flexibility and porosity. We have also found that this, until now, could only be done with the original binder, lime. The only additives that, at least to date, have worked well are those giving higher porosity. Replacing part of the natural sand (30-100%) with crushed dolomite (calcium-magnesium carbonate) has improved setting and hardening. It also raises the total amount of carbonate, which improves resistance to acidic pollution because it acts as a buffer. As the dolomite particles are irregular in shape, with rough surfaces compared to natural sand, the workability of the fresh mortar is somewhat affected. Not having much more than ten years of experience, we do not know much about the long term ageing of the dolomite mixtures.

Reproducing old mortars

As mentioned above, we try to make the mortars for repair with the same mechanical strength and moisture properties as the original. The problem is that we know in fact very little about these properties. We have no reliable figures and tests on the original mortars, and rather vague figures on the new material. Thus we try to make the new mortars from the same recipe as the original (same kind of lime, same sand, same proportions) and after some on-site tests, try to see whether the properties are comparable. This is a rough and very uncertain way of doing it. A recipe is really no guarantee for a specific result either in strength or in porosity. The process of mixing, of working and treating the material all through the process affects the final product, i.e. the rendering or the masonry joint. For example, the choice of the limestone, the burning and - last but not least - the slaking process affect the properties of the lime as a binder. Standard chemical tests on the slaked lime may show no difference, but in practice, differences in its behaviour can be like night and day.

Lime mortar is really a curious product, hard to understand fully. Having worked with these problems both theoretically, in the laboratory, and in the field more or less intensely for some twenty years, I think the problem can be solved only by interdisciplinary teamwork. Specialists in geology, chemistry, testing techniques, mechanical strength, etc., have to work together with historians, archaeologists, ethnographers, etc., as well as architects, building engineers and, perhaps most important, masons. And when I say teamwork I really mean a team where the specialists work <u>together</u> physically at the building site, in the laboratory, in the quarry, to be able to understand the whole context and to be able to feed each other with questions and ideas. An international symposium is one good start to such teamwork.

To be able to make good mortars for repair, we need to know the properties of the original ones being replaced and how to test these properties. With that knowledge we would be able to construct a new mortar with the desired properties. Today this is not possible; we can only make mortars from the same or - perhaps more adequate - similar ingredients as the original. Mixing these in one way or another we can only hope that the properties will be the same as the original. Sometimes we seem to succeed, but we cannot be sure. Sometimes we can easily see that we fail.

This is of course too uncertain to be satisfactory. We have to improve reliability in the work process. Today it is too much like a game of chance. One good thing in this uncertain situation is that at least if the new lime mortar fails, it never harms the building, the original. That is one of the major criteria of a suitable material for preservation: it must never damage the original. The addition, the lime mortar, can always be removed, if it also fails in some other way, without doing any harm to the original. It is always weak enough. So we still say that lime mortar should be replaced with lime mortar, and on historic buildings portland cement should not be used.

Research work to be done

According to my experience there are some urgent investigations to start with:

1. measure the mechanical strength of the original mortars;
2. measure the moisture properties of the original mortars.

This could seem to be rather simple, at least if we do not intend to measure all mortars used in the past. The problem is that there are no suitable standard tests for either of these purposes. For example, tests of compressive strength as well as tensile strength require samples of so great a size that it is almost impossible to take them out of a masonry joint, a rendering or whatever. If we use smaller samples we do not know how reliable they are and how to compare them with standard ones. We also need testing methods that can be used in the field, on the building site. They should also, if possible, be non-destructive.

Moisture properties are still more difficult to test. We do not even know what to test. The standard tests on diffusion and of water absorption give no adequate description of the moisture properties. Perhaps we should still use them to be able to compare with materials already tested. Here, too, we have the problems of the size of the test samples. I think it is worth trying another line of attack on the question of moisture properties. In mortars consisting of mineral materials, the moisture properties vary almost entirely with the pore structure. If we can study this structure (pore size, distribution, total amount, and interconnections), we could directly compare one mortar with another. This information is sufficient and very precise when one is trying to find a duplicate to an existing mortar. The size of the test sample is not critical, which is really an advantage.

Apart from the problem of describing the technical properties of existing mortars, we have the testing of new mortars. The standardized sample for mechanical tests, a prism 30×70×150 mm, should be cast in a steel mould and stored in a certain humidity for a specified time. The whole procedure is unsuitable for mortars based on quick lime. They do not harden, they only dry out. The samples are too thick and too wet to carbonize. To that is added the fact that there can be too little carbon dioxide present in the surrounding air. The conditions are highly dissimilar to those for an external rendering on a brick wall. The procedure of making the test samples is, on the other hand, nearly perfect for concrete and mortars based on portland cement and usable for other hydraulic binders. Comparative tests between quick lime, portland cement, and other hydraulic binders are thus false, useless, if made in the standardized way.

The mortar based on quick lime must be cast in a mould with a certain suction in order to remove excess water (like a wall!), and should be allowed to dry under conditions of "normal" external humidity surrounded by moving air with a normal CO_2 content. Samples should eventually be dipped in water (or sprayed), imitating the treatment they would receive on a normal building site during the hardening process.

I propose the following factors for investigation.

A. Mechanical properties of old mortar

1. Compressive strength
2. Tensile strength
3. The modulus of elasticity

(4. Adhesion)

B. Moisture properties of old mortar

1. Pore structure (amount, size, distribution, connection)
2. Water absorption

To this are added chemical tests, microscopy, etc., to characterize the mortar: type of binder, sand, additives, proportions, etc.

To be able to investigate A and B above, we have to find new methods of testing - or at least modify existing methods. These methods must include techniques for taking samples (sawing, core-drilling, etc.). With regard to the problems of taking samples from a valuable historic fabric, we should also find at least some non-destructive method that can be used in situ.

Parallel to the development of testing methods for existing mortar, old mortar, we have to modify the methods for corresponding tests on new mortar. In particular, the way of mixing, casting, and treating the mortar to get the samples must be studied carefully. I doubt that quick lime and hydraulic lime should be treated in the same way, but in any case they should not be treated like portland cement because the hardening process is entirely different. Existing standardized methods are designed for portland cement.

Knowledge gives new possibilities

When we are able to describe the main functional properties of a mortar we can start to reconstruct that mortar or construct another with the desired properties. This is not possible today. We can only reconstruct the compositions, and hope that they will work.

By checking the functional properties of a new mortar of a certain composition (reconstructed old or entirely new), we can also study how other factors like the mixing method or application after treatment are influenced. That also means we then have possibilities to test theories or vague information about, for example, a mediaeval mixing method, a special way of slaking, or the like. We can compare the resulting physical properties of a reconstructed mortar with an old one of identical composition.

Adequate methods for testing mortars in historic buildings are of interest also in the general building market because when repairing the rendering or masonry of an ordinary building it is also important for the new material to cooperate with the original. Such cooperation is guaranteed only if the materials are similar in their technical behaviour The development of test methods as described above ought in fact to be of great interest to the whole building market and especially to the building research organizations.

ORGANICS VS. SYNTHETICS: THEIR USE AS ADDITIVES IN MORTARS

LAUREN-BROOK SICKELS

SUMMARY

Records survive that show that such organic additives as blood, cheese, egg, and dung were used in mortars made in past centuries. Restorationists may face the question of whether to replace old mortar with similar organics or use more technologically advanced components such as synthetics. Preliminary research was conducted on the various uses of organic and synthetic admixtures. Simultaneously, weathering and exposure tests were conducted on a variety of organics. The compiled results and factors for and against the use of the two types of additives are set forth in this paper.

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24 July 1981

Definitions

'Organic' and 'synthetic,' for the purposes of this paper, have meanings somewhat different from strict dictionary definitions. Organic is a term used to describe all materials found in a natural state. These items are usually unadulterated before being combined with other materials in a mortar. However, they may be modified (e.g. liquidized with water, or strained) before use. Synthetic is a term used to describe both man-made materials and natural materials which have been adulterated or pretreated in some way before being employed in a mortar. An organic additive, as defined above, can become a 'synthetic' simply by altering its natural state.

Objectives

This paper has three main objectives: 1) to inform the reader of potential alternatives to organic additives in mortars, and the pros and cons of their use; 2) to present the results of a preliminary research and testing program being conducted on lime mortars at the University of Edinburgh; and 3) to provide tables on synthetic materials suitable for potential use and a list of suppliers, whereby the reader can deduce which materials might fit a certain project, and obtain them.

Introduction

Lime and sand were not the sole constituents of mortar before the advent of Portland cement. Masons of centuries past were continually trying to improve mortars. The Romans were known for their use of pozzolanic elements and in certain regions of Holland, Germany, and Great Britain equivalents existed. Availability and cost were large factors, and therefore, not all masons had these constituents at their disposal. Other sources had to be found to impart the necessary and desired properties to an often-weak lime and sand paste. Organic admixtures such as blood, egg, sugar, cheese, and dung offered the mason a variety of solutions. Extensive documents survive to inform historians of their wide and continual use since as early as the last centuries B.C.(Sickels, 1981) Table I lists many of the organic admixtures written about throughout history, and their dates of introduction and subsequent use.

One problem facing restorationists today is: how to restore mortars. Does one analyze them and replace the old with the same materials, or does one use modern synthetics and match the old as closely as possible?

Early organic additives

Masons had to rely on experimentation or information passed on orally to understand and learn of the properties of organics and the effects they would produce when added to a mortar. Occasionally, literature might be available. Today, restorationists are more fortunate in that analyses of old mortars, plasters, and other ancient materials have been conducted in recent decades, the results of which have been published to further contribute to the understanding of organic admixtures.(Neuburger, 1930; Hodges, 1964) Albert Neuburger discovered that gum arabic or tragacanth, animal glue from Rhodes, the blood of hippopotomus and the milky juice of figs mixed with egg yolk all served the ancients as adhesives or binding substances.(Neuburger, 1930) Egg albumen, keratin, and casein were other common Egyptian natural organić polymeric binders.(Sayre, 1976) In Vitruvius's time, fig juice, rye dough, hogs' lard, curdled milk, blood, and egg whites were employed to toughen and regulate setting qualities.(Bankart, 1908) Blood and egg whites are also mentioned as having been used to retard the set.

Upon obtaining a basic knowledge of the organics' properties, an understanding can be reached as to why they were used in various projects of past centuries. For example, in the ninth century A.D., Queen Eleanor's cross at Charing Cross, London, was erected using the white of eggs and the strongest wort of malt mixed with lime and Calais sand.(Bankart, 1908) At the end of that same century, bullocks' blood was mixed into the mortar for Rochester Cathedral and later still, during the sixteenth century, urine was used at Rouen Cathedral.(Bankart, 1908) The egg whites and blood acted as adhesives and also retarded and regulated the set. The malt and urine acted as air entrainers to improve the durability of the mortar with their crystalline sets.

As some early lime mortars had very little strength, it was not uncommon to find fibrous plant materials or coarse animal hairs mixed in for greater strength.(Hodges, 1964) For example, at Justinian's Church of the Baptist, Constantinople, elm bark and hot barley water (tannin and size) were mixed into the stucco mortar.(Bankart, 1908)

Based on this research and the conclusions drawn, a table was compiled to compare the various properties of some organics and synthetics. Table II gives, for each kind of admixture, organics which carry these traits and their synthetic equivalents. It also enables restorationists to understand the use of the organic and helps him determine which one would suit his use if he used one or more in a new mortar.

The question still stands, however, as to how and why synthetics offer a solution or alternative to the use of organic additives in mortar. The main, obvious problem with organics is the inability to control the results they create on contact with various building materials and on exposure to weather and biological growth. Organics create several other problems which must be overcome before they are truly suitable in a restoration mortar. Tests of some organic components showed their limitations, thus ruling them out in favor of more advanced, controllable, synthetic materials.

Some deficiencies and limitations in the use of organic additives

In May 1981 weathering and exposure tests were run on twelve different organic mortar patties. This testing was done: 1) to see if the organics, indeed, did have the properties the ancients said they had; and 2) to determine how effective and durable they would be if used in restoration projects. Each patty was made either by following a surviving mortar recipe or by using a 1:3 lime:sand mortar recommended by Vitruvius and other scholars and adding various organics to this base. The patties were aged eleven days before they were tested. Table III gives a description of each mortar's components and proportions and how well they performed during the tests. At the same time, extensive research was conducted to find results from other organic tests and to understand the chemistry of each of the components in each of the organics. Conclusions were then drawn based on the research and tests.

Briefly stated, organics cannot be controlled easily due to the nature or chemistry of the various components. As they are natural materials, other admixtures would have to be introduced to alter these existing admixtures; but there is an easier and simpler solution due to the technology we possess today, one which will enable the same effects produced by an organic admixture to take place. The answer: synthetics.

A few of the problems discovered during the testing of the organics and the concurrent research were biological attack, solubility, and the inability to produce the desired property. Attack from biological organisms was the largest problem that arose. One test was to leave all twelve mortar patties outdoors for 24 hours. Ants, flies, and ground squirrels immediately took an active interest in the patty containing ox blood, and in the one consisting of cheese, milk, and eggs. If given a chance, lichens also would have begun to grow.

Lichens are encouraged by any organic with a large nitrogen content, so mortar made with urine or manure will attract microörganisms. Within the latter two organics, nitrogen is found in the form of ammonia. The ammonia is absorbed into the mortar and ultimately into any surrounding porous building materials. <u>Nitrosomonas</u> bacteria are present and convert the ammonia to nitrites, after which <u>Nitrobacter</u> convert the nitrites to nitrates. The nitrates set off a chain of events which can eventually lead to spalling, cracking, or popping mortar. This entire process beginning with the simple use of animal excrement proves that more controllable alternatives should be sought.

Solubility in water is another problem. Hot barley water or tannin and size were recommended by the ancients as strengtheners. Tannin, in particular, had the power of converting animal skins into a very durable substance. However when used alone, it has proven to dissolve in water. An ordinary cup of tea can be used as one common example of the ease with which tannin dissolves. Heaven help the occupant whose house with tannin-rich mortar sank around his ears after a few rainfalls. An alternative to soluble organics needs to

Figure 1: The twelve mortar patties were photographed before testing. Chunam stucco, in the upper left corner, had to be withdrawn from testing as it never solidified.

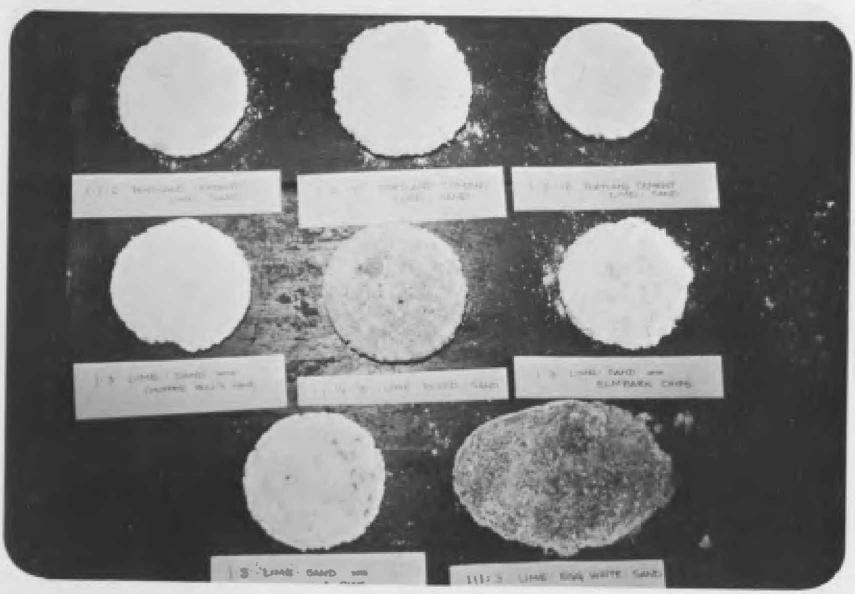


Figure 2: These eight patties were made from mixes mentioned in literature. These mixes appeared to be used more frequently than those of the remaining four patties.

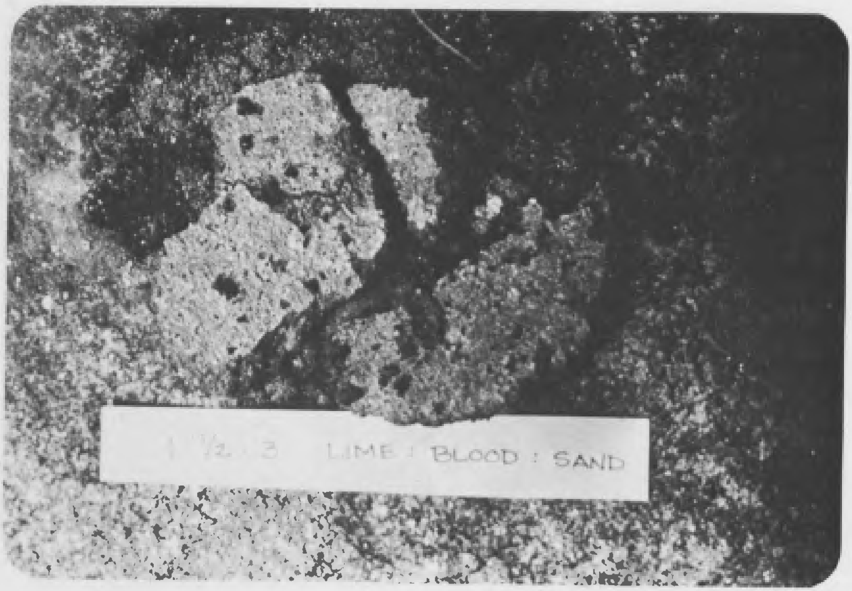


Figure 3: The patty containing blood broke into several pieces immediately after exposure to a one-minute rainfall.

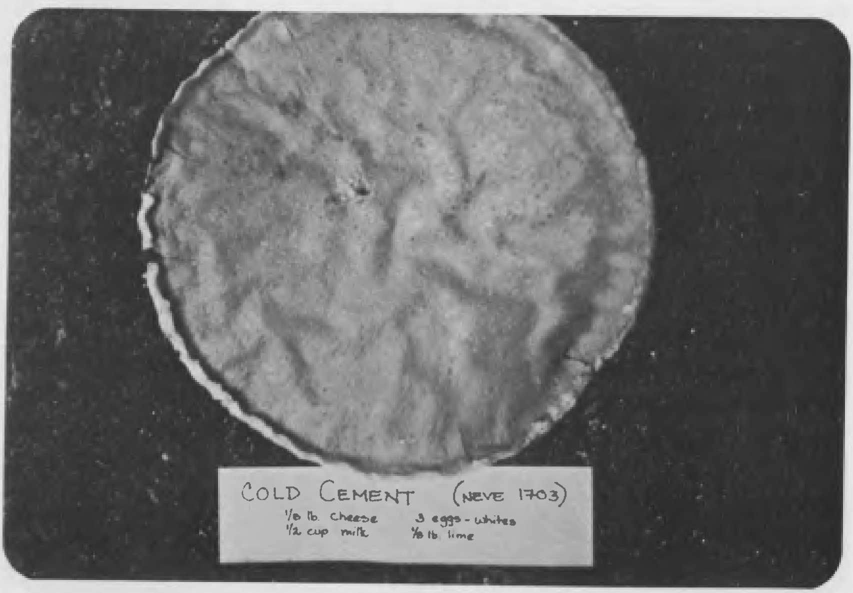


Figure 4: Although ants and ground squirrels showed an interest in the Cold Cement patty, it held up very well under testing. While the surface is irregular, the patty resembles rubber in texture and has excellent plastic qualities.

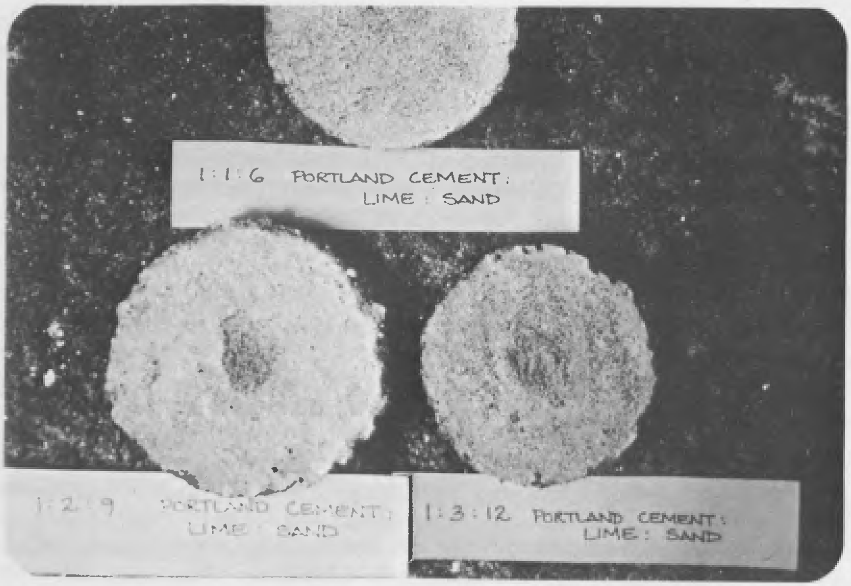


Figure 5: These were the only patties made using Portland cement. The proportions for each mix were obtained from literature and are now used by several British Government organizations.

be sought.

A similar case can be argued against the use of blood. Pliny the Elder, in his books on natural history, elaborated on the use of blood and its excellent qualities, particularly as a strengthener. He said bullocks' blood "coagulates and hardens the most speedily of all." Mortar patty No. 6, consisting of lime, sand, and bullock's blood, however, did not withstand one wet-dry cycle before it broke into several large pieces. For more reliability, a synthetic admixture could prove more successful.

Under preliminary testing, organic mortars have not produced favorable results. Poor performance during and after the outdoor exposure and weathering tests lead one to ask the question: why not use synthetics as replacements? The issue now becomes one of <u>which</u> synthetics offer alternatives to a particular organic.

Synthetics

Synthetics are relatively new to the building industry, gaining an increased popularity in the last two decades. They can serve in the form of an admixture to a mortar base or as the main components in caulks and sealants. The most favorable aspect of these artificial materials is that they can be controlled more than the organics. Synthetics can often be combined with others of their kind to create a product with the best qualities of each of the individual components, or they can be used alone to impart desired properties. The drawbacks of the use of synthetics initially appear to be minor and surmountable, making these artificial materials more favorable then organics if caution and care are observed.

Epoxy polyester is an excellent example of a combination of synthetics and can be used, in connection with a mortar, as an adhesive, an admixture, or a protective surface coating. Each of the two components can be varied to achieve required characteristics. By increasing the amount of polyester, solvent and acid resistance can be increased. The former property makes polyester a suitable alternative to tannin. Toughness and hardness can also be regulated. However, by increasing the epoxy portion, gloss retention, flexibility, adhesion, and alkali resistance are enhanced. Furthermore, the setting time of epoxy polyesters can be controlled by regulating the two components.

Acrylics and polyvinyl acetate (PVA) have also showed considerable promise in the field of mortar admixtures as single components, not necessarily requiring an additional constituent. Among the various synthetics available as alternatives to organics, these two carry the qualities of color stability, stability over a wider range of temperature, and greater strength. One drawback is that acrylics tend to be expensive, more so than PVA. Acrylic emulsions are, nevertheless, widely used, and their success over the organics in mortars is apparent.

Earlier it was shown how blood had proven to be an unreliable organic additive. Acrylics have similar characteristics as those the ancients believed blood to have. Through research this has been substantiated.(Lavelle, 1980?) Rohm and Haas Company, U.S.A., tested the durability of latex-modified cement mortars using acrylic, styrene/butadiene, and vinyl acetate as modifiers. Where blood-rich mortar could not tolerate exposure to a one-minute rainfall, the acrylic-modified mortar proved to increase its strength the longer it was exposed to weathering. Figure 6 shows the results graphically. Based on results such as these, Table II and others like it can be compiled to give restorationists a quick reference when faced with a mortar project involving organic additives. Table IV goes one step further and gives a few examples of problems caused by organics and how certain synthetics offer favorable solutions.

In addition to research on the beneficial use of synthetics, Rohm and Hass also manufactures acrylic emulsions, particularly Rhoplex[R] LC-67, a binder, and Rhoplex[R] MC-76, a modifier. Like epoxy polyester and other two-part synthetic systems, these two products impart qualities which allow a mixture of them to be used as an adhesive, a plasticizer, a filler, and a strengthener. The property is achieved based on the quantity of each Rhoplex used. Again, it is this control, versatility, and reliability which are tending to make synthetics more popular than original organic admixtures.

Drawbacks

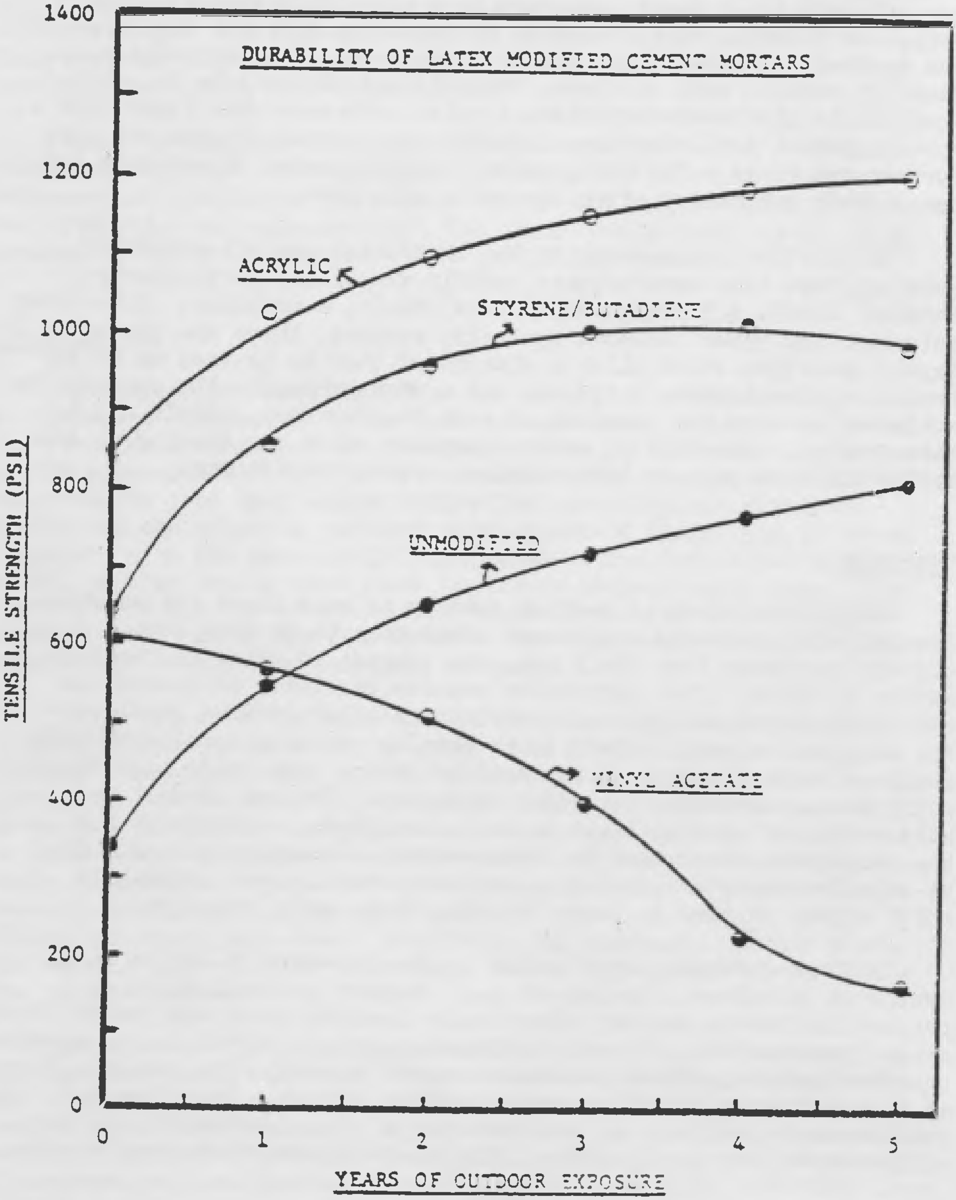
Only a few words of caution need to be made about the possible drawbacks of synthetic admixtures other than their high cost. Side effects can occur from their use, the largest of which is the formation of salts. This particular residue can lead, of course, to efflorescence, spalling, and a variety of other serious problems. For example, Acrysol[R] ASE-60 is an acrylic emulsion copolymer which requires neutralization of its acidity with a base prior to acting as a thickening admixture for other emulsions. The end product is a salt which, if used in large amounts, can increase shrinkage due to the additional water that is incorporated. Table V contains a list of selected admixture qualities and some organics and synthetics which might, if used in large amounts, cause salt formation.

A final drawback worth noting is the irreversibility of many synthetic additives. Emulsions that require neutralization or preparation before use may alter their chemical state and render themselves irreversible. The aforementioned Acrysol ASE-60 is one example. Once neutralized, ASE-60 solutions cannot be reverted to emulsion form as a reduction in pH will precipitate the polymer. Suppliers and manufacturers can help to overcome some of these drawbacks, as they will know whether their product fits specific admixture requirements.

Case study

Although mortar is generally considered to be an exterior building product, masons of past centuries considered plaster and

Figure 6: Synthetic Additives in mortars. From J. A. Lavelle, "Cementitious Coatings," paper for Rohm & Haas Co., 1980(?).



interior stuccoes to be just watered or slurried versions of the mixes used outdoors. This opinion has been verified as the materials used by the Society for the Preservation of New England Antiquities/ National Park Service at St. Johnsbury Academy, St. Johnsbury, Vermont, U.S.A., in mixing new plaster could also be used in exterior mortars.(Phillips, 1980) One useful plaster formula imparting flexibility and workability was recommended as follows:

- 2 parts lime
- ½ part water
- 2 parts fluid coke
- 3 parts of a mixture of 50% Rhoplex[R] MC-76, 50% Rhoplex[R] LC-67
- 2 parts Eccospheres[R] IG-101 microballoons
- thickener, Acrysol[R] ASE-60 or Cab-o-sil[R] M-5, as desired.

A base mix traditionally consists of lime, sand, and water. Here the mix calls for only lime and water. The sand component has been eliminated in favor of a synthetic, fluid coke, that contains the same properties as sand and yet offers more. The coke is used as a filler and a shrinkage compensator, therefore serving as both a component of the base mix and as an admixture. The rest of the recipe can be labelled synthetic admixtures. The Acrysol and Cab-o-sil are thickeners, as noted. The two Rhoplex products serve as binders, and the microballoons are used as fillers to help reduce the amount of liquid binder needed and to minimize drying shrinkage. Product literature from the various manufacturers of these synthetics shows that the constituents can also be employed in mortars used outdoors.

Conclusion

The market for synthetics is continually expanding. New are being created while the old are perfected. Preliminary tests have shown that a lime mortar with an organic admixture can create problems which could mean future repairs. Such organics as blood can not be controlled. Effects such as color and staining would have to be accepted with the hope that they would match the old, assuming first that the organic chosen could withstand weathering. Synthetics, on the other hand, have shown considerable promise in the field of admixtures due to the control the user has over them. While caution needs to be observed and the quantities regulated to avoid such side effects as salt formation, synthetics nevertheless are proving favorable in the properties they impart. Many of them are optically clear in nature and would not disturb the original color and appearance of the mortar. It would be desirable to find a synthetic alternative that would contain all the desired effects the mortar previously had while introducing no further problems. The result would be a mortar able to match the old mix and withstand many more decades of weathering.

A restorationist is clearly faced with a philosophical issue: how to recreate the old mortar and yet achieve certain qualities so as to meet the demands of today's construction industry. Furthermore, we are faced with standard codes and requirements. The American Society of Testing and Materials in the U.S.A. and equivalents elsewhere establish minimum building standards. Most organics, being fairly unreliable, may be eliminated on this factor alone. Synthetics are proving reliable in the field of building construction.

Considerable further work, both in research and in testing, needs to be done on organic and perhaps more importantly synthetic admixtures. But to date it appears that if the question of how to restore organic mortars should be rephrased to ask: which synthetic best meets the qualities and demands originally met by the organic admixture, or what synthetic is its equivalent?

Table I: List of organic materials and their dates. From L. B. Sickels, "Organic Additives in Mortars," in E. A. R., Vol. 8, 1981, p. 15.

	150 BC Egyptian	46 BC Vitruvius' time	23 AD Pliny	800 AD Rochester Cathedral	1200	1500 Middle Ages	1653 Plat	1703 Neve, Moxon	mid-1700s	1837 Vicat, Smith	1850 Burnell & periodicals
albumen	X										
animal glue	X							X			X
barley			X						X		
beer					X	X			X		
beeswax					X	X		X	X		X
blood	X	X	X	X		X	X	X	X		X
butter										X	
buttermilk									X		
casein	X										
cheese								X	X	X	X
cotton											X
curdled milk		X							X	X	
dung									X		
eggs	X				X	X		X	X		X
egg whites	X	X			X	X	X	X		X	X
elm bark			X								
fibers			X								
fig juice	X	X	X						X		
fruit juices					X	X			X		
gluten					X	X			X		
gum arabic	X					X	X				X
hair			X								
hogs' lard		X	X					X			X
keratin	X										
malt					X	X					
milk		X	X					X	X	X	X
molasses										X	
oil			X							X	X
resin								X			
rice					X	X					
rye dough		X							X		
saffron			X								
shellac											X
size			X		X	X					
suet			X								
sugar					X	X				X	
tannin			X								
urine					X	X					
vegetable juice									X		
wine			X								
wort					X				X		

Table II: A list of selected admixtures for possible use in mortars (and plasters).

Type of admixture	Desired effect	Organic material	Synthetic material[a]	Example
accelerator	accelerate setting	egg whites	calcium chloride	
	and early strength	elmbark	triethanolamine	
	development	barley water	calcium formate	
		fig juice	epoxies	
		rye dough	urea with barium	
		hogs' lard	hydroxide	
		curdled milk		
		blood		
		starch		
		colostra		
		gum mastic		
		sugar		
adhesive/	increase bond	rosin	acrylic resins	
tackifiers		modified rosin	acrylic polymer	Acryl 60
		gelatin	acrylic polymer	
		animal glues	emulsion binder	Rhoplex[R] LC-67
		esp. hides	acrylic emulsion	Rhoplex[R] MC-76
		gluten	epoxy polymer	Durabond EP
		vegetable glues	rubber	
		casein	butadiene-styrene	
		blood albumen	copolymers	
			polyvinyl chloride	
			(PVC)	
			polyvinyl acetate	
			(PVA)	
			PVA with PV alcohol	Rendabond

[a] Some of these synthetics may need an additional synthetic in order to impart the required properties (e.g. air entrainers).

Table II cont.

Type of admixture	Desired effect	Organic material	Synthetic material	Example
air entrainer	improve durability	malt beer urine animal hides (salts of proteinaceous materials)	alkyl-aryl sulphonates some synthetic detergents salts of lignosulphonates alkylbenzene sulphonates petroleum fractions barium hydroxides sulphonated hydrocarbons	
emulsifier/ stabilizer	stabilises an emulsion (use in small quantities)	egg yolk oils fats waxes	benzophenones[b] benzotriazoles acrylonitriles	
filler	improve hardness	size glue gum arabic talc sugar saccharine fruit juices gluten rice	coarse fluid coke acrylic polymer emulsion binder acrylic emulsion sodium borosilicate glass	 Rhoplex[R] LC-67 Rhoplex[R] MC-76 Eccospheres[R] IG-101
frost resistor	resistance against frost penetration	sugar	polythene acrylics propyl alcohol	

[b]These synthetics are used to stabilize an emulsion against ultra-violet light; they are UV absorbers.

Table II cont.

Type of admixture	Desired effect	Organic material	Synthetic material	Example
gas former	causes expansion on setting	vegetable glue	resin soap	
		animal glue	hydrolized protein	
			aluminum powder	
			saponin	
modifiers	alters existing solution	proteins	some acrylics	
		egg whites	acrylic polymer	Rhoplex[R] E-330
		hemp seed	acrylic emulsion	Rhoplex[R] MC-76
		blood	acrylic powder	Rhoplex[R] LC-67
		gluten	polyvinyl acetate (PVA)	
		keratin	water-soluble resins	
		collagen	formaldehyde	
		casein	cellulose derivatives	
		gelatin	triethylene glycol	
		borax	glycol ester	
		natural resins	styrene/butadiene copolymers	
			pyrogenic silica	Cab-o-sil[R] M-5
non-shrinking agents	prevents shrinkage	beeswax	fluid coke	Five Star Grout
			aluminum powder	
			silica gel	
			acrylic polymer emulsion binder	Rhoplex[R] LC-67
			acrylic emulsion	Rhoplex[R] MC-76

Table II cont.

Type of admixture	Desired effect	Organic material	Synthetic material	Example
plasticizer/ mollifier	impart plasticity reduce brittleness; a softener increase workability	sugar milk egg whites slurried dung Turkey red oil animal glue: glycerine glucose soyabean lecithin mineral oil rosin non-drying oils: linseed oil hogs' lard figs	hydroxylated carboxylic acids salts of lignosulphonates silicones Vinsol resin or phenol-formaldehyde resin[c] sodium borosilicate glass epoxies phosphates glycolates polybutenes phthalates acrylic emulsion acrylic polymer emulsion binder	Cemplas Rendaplas Eccospheres[R] IG-101 Rhoplex[R] MC-76 Rhoplex[R] LC-67
retarder[d]	retards setting time	sugar blood egg whites saccharine gluten borax	lignin tartaric acid and salts silicones hydroxylated carboxylic acids	

[c]Vinsol resin is also an extender.

[d]Some retarders may carry air entrainers or have the properties known to air entrainers.

Table II cont.

Type of admixture	Desired effect	Organic material	Synthetic material	Example
solidifier/ rigidifier	increase hardness or stiffness	sugar vegetable glues[e] animal glues treacle molasses	silicones silane coupling agent baryta polyurethanes	 Brethane Stone Preservative
solubiliser/ dissolver	prevention of growth of micro-organisms		borate tributyltin oxide with quat quaternary ammonium compounds sodium methyl siliconate[f] with sodium salt of phenol (e.g. penta-chlorophenol or ortho-phenylphenol)	
strengthener/ binder	improves strength of solution	keratin casein elmbark hot barley water tannin size linseed oil walnut oil cow/ox/human hair chopped straw rice rye dough[g]	some acrylics acrylonitrile acrylic emulsion acrylic polymer emulsion binder nylon fluid coke polyvinyl chloride (PVC) polyvinyl acetate (PVA) polyethylene terephthalate polythene propyl alcohol	 Rhoplex[R] MC-76 Rhoplex[R] LC-67

[e] Upon exposure to the sun, vegetable and animal glues become rigid.
[f] Sodium methyl siliconate with sodium salt of phenol may cause staining.
[g] The use of rye dough requires a non-shrinking agent.

Table II cont.

Type of admixture	Desired effect	Organic material	Synthetic material	Example
strengthener cont.		egg whites fibrin in blood cotton flock jute sisal gum arabic or tragacanth animal glue from Rhodes fig juices with egg yolks sugar		
superplasticizer	high flow	albumen	melamine formaldehyde sulphonates naphthalene formaldehyde sulphonates	
thickener	thickens the consistency of an emulsion	blood sour milk casein cheese collagen gelatin gum tragacanth with water	pyrogenic silica acrylic emulsion copolymer	Cab-o-sil[R] M-5 Acrysol[R] ASE-60
waterproofing, dampproofing and weatherproofing repellent	decrease permeability	animal glue plus tannin bitumen wax emulsion	soluble chlorides calcium stearate aluminum stearate ammonium stearate butyl stearate methyl groups	

Table II cont.

Type of admixture	Desired effect	Organic material	Synthetic material	Example
waterproofing, etc. repellent cont.		mineral oil emulsion beeswax	stearic acid oleic acid polyurethanes[h] polysylphide sealant	 Tremco Lasto-Meric
			epoxy resins with silicones	Sealocrete products
			epoxy-terminated polyurethanes silicones	Tremco DYmeric
			silane coupling agent	Brethane Stone Preservative
			silicone resin with mineral spirits sodium methyl siliconate with water polyvinyl acetate (PVA) fluid coke acrylic resins	
water reducer[i]	reduces water required for given consistency	sugar	salts of lignosulphonates hydroxylated carboxylic acids gluconates heptonates polysaccharides tartrates citrates	

[h]Polyurethanes are generally replacing polysylphides.
[i]Water reducers tend to retard set, so accelerators should be added. Some water reducers may also cause shrinkage.

Table II cont.

Type of admixture	Desired effect	Organic material	Synthetic material	Example
water reducer cont.			silicones melamine formaldehyde sulphonates naphthalene formaldehyde sulphonates	

Table III: Results of tests on organic additives in mortar.

Mortar description	Resistance to shrinkage	Wet/Dry cycle	Protection from biological attack	Quality of appearance
1. "Cold cement" (c.1703): 1/8 lb. cheese, 1/2 cup milk, 3 eggs, 1/8 lb. lime	E	E	F	E
2. "Chunam stucco" (c.1837): 3 egg whites, 1/8 lb. butter, 1 cup milk, 1/16 lb. lime	VP	X	F	VP
3. 1:1:3 Lime:bone meal:sand	G	G	G	G
4. 1:3:12 Portland cement:lime:sand	E	E	E	E
5. 1:1:6 Portland cement:lime:sand	F	E	E	E
6. 1:½:3 Lime:bullock's blood:sand	G	VP	VP	P
7. 1:3 Lime:sand with elmbark	F	G	G	F
8. 1:3 Lime:sand with bull's hair	G	G	G	G
9. 1:3 Lime:sand with wild rice	G	F	G	G
10. 1:1:3 Lime:egg white:sand	P	F	G	P
11. 1:1:3 Lime:vegetable juice:sand	F	G	G	F
12. 1:2:9 Portland cement:lime:sand	E	E	E	E

KEY:

Excellent.........E
Good..............G
Fair..............F
Poor.............P
Very Poor.........VP
Not Applicable....X

Table IV: Selected examples of synthetic alternatives.

Type of admixture	Organic material	Problem	Synthetic solution
adhesive	rosin	yellows	epoxy polymer, Durabond EP
air entrainer	urine	lichen growth	polyethylene oxide
plasticizer	linseed oil	low film strength at low temperatures	acrylate copolymers PVA
strengthener	tannin	soluble in water	acrylic emulsions, Rhoplex[R] MC-76 Rhoplex[R] LC-67
thickener	blood	red or dark stain attracts insects and rodents	pyrogenic silica, Cab-o-sil[R] M-5
waterproofer	casein	not outstanding resistant to hydrolysis, oxidative deterioration or microbiological attack	silicones silane coupling agent, Brethane Stone Preservative

Table V: A selected list of admixtures which may undergo chemical changes. From M. R. Rixom, Chemical Admixtures for Concrete (London: E. & F. N. Spon, Ltd., 1978), pp. 218-19.

Type of Admixture	Chemical Name	Possible Chemical Changes
accelerators	calcium chloride	partial formation of chloroaluminates
	calcium formate	partial formation of formoaluminates
	triethanolamine	complex formation with C_3A hydrates
air entrainers	tall oil soaps	calcium salt formation
	short chain fatty acid soaps	calcium salt formation
	neutralized wood resins	calcium salt formation
	alkyl-aryl sulphonates	calcium salt formation
waterproofers	calcium stearate	none
	stearic acid	calcium salt formation
	oleic acid	calcium salt formation
	wax emulsion	none
	mineral oil emulsion	none
	butyl stearate	partial hydrolysis
water reducers	lignosulphonates	calcium salt formation
	gluconates	calcium salt formation
	heptonates	calcium salt formation
	polysaccharides	calcium adduct formation
	melamine formaldehyde sulphonates	calcium salt formation
	naphthalene formaldehyde sulphonates	calcium salt formation
	tartrates	calcium salt formation
	citrates	calcium salt formation

Appendix I: Organic additives with high levels of nitrogen and potentially subject to attack by lichens.

Organic name	% Nitrogen
albumen	15.70
barley	12.96
casein	16.60
collagen	17.86
egg whites	20.40
gelatine	18.30
milk	8.00
rice	6.30
urine	1.42 (95% is water)

Appendix II: Names and suppliers for synthetic products mentioned in this article.

Name of synthetic	Supplier
Acryl 60[R]	Thoro System Products 7800 N.W. 38th Street Miami, Florida 33166 U.S.A.
Acrysol[R] ASE-60	Rohm & Haas Co. Independence Mall West Philadelphia, Pennsylvania 19105
Acryloid[R] MC-46	Rohm & Haas Co., U.S.A.
Brethane Stone Preservative	Building Research Establishment Garston, Watford WD2 7JR England
Cab-o-sil[R] M-5	Cabot Corp. 125 High Street Boston, Massachusetts 02110 U.S.A.
Cemplas	Cementone Ltd. Tingewick Road Buckingham MK18 1AN England
Durabond EP	MBS Resins Ltd. West Carr Road Industrial Estate Retford, Notts. England
Eccospheres[R] IG-101 microballoons	Emerson & Cuming Canton, Massachusetts 02021 U.S.A.
Elvace	DuPont de Nemours 350 Fifth Avenue New York, New York 10001 U.S.A.

Appendix II cont.

Name of synthetic	Supplier
Elvanol	DuPont de Nemours
Elvax	DuPont de Nemours
Five Star Grout	U. S. Grout Corp. West End Avenue Old Greenwich, Connecticut 06870 U.S.A.
Hypalon	DuPont de Nemours
Neoprene	DuPont de Nemours
Rendabond	Cementone Ltd.
Rendaplas	Cementone Ltd.
Rhoplex[R] E-330	Rohm & Haas Co.
Rhoplex[R] LC-67	Rohm & Haas Co.
Rhoplex[R] MC-76	Rohm & Haas Co.
Sealocrete products	Sealocrete Products Ltd. Altantic Works, Oakley Road Southampton S09 4FL England
Tremco DYmeric	Tremco Ltd. 46 Arrotshole Road College Milton North East Kilbride G74 5DN Scotland
Tremco Lasto-Meric	Tremco Ltd.

Restorationists:

Peter Cox Ltd.
Wandle Way
Mitcham, Surrey CR4 4NB
England

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LIME WATER CONSOLIDATION

STEN PETERSON *

SUMMARY

The article describes the method of consolidation with lime water. This traditional method has been used by restorers and craftsmen for centuries with good results.

The method works by replacing calcium in brittle or friable plaster or mortar, or in flaking or powdering paint layers. In other words, the binding medium in mortar, plaster or pigments is regenerated.

This consolidant is not an addition of a compound of a different character into the original materials. This means that the consolidated material retains its original properties and behavior.

* Restorer and member of the Nordic Group of IIC

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INTRODUCTION

Today the chemical industries have developed many materials, mostly of organic character, for the consolidation of works of art. For these chemicals long life, reversibility and no discouloring are promised. These promises are mostly based on laboratory tests, carried out during a very short time compared with the age of the material in a historic building or a work of art, which are in general placed in an environment with hard changing conditions for centuries. To test a method in situ is always the best but slow. The traditional methods are developed during a long time and the time is the only way to prove if a method is good or not.

Nowadays restorers are working hard all over the world to clean and save wall paintings, conserved in the past with shellac, wax or casein. The solubility and reversibility of these materials are reduced by the time. The restorers are often forced to use solvents which could damage the paintings or the plaster. Are we sure that it will be possible to restore, or to save our works of art in the future if we are using a modern synthetic consolidant? Probably the future restorers will meet quite a lot of problems in removing consolidants which we are using today.

During the development of the new building industries in our time, a decadence of the traditional technical knowledge and skilled craftmanship of builders and artisans has occured. It is urgent, as well as our duty, to preserve as many as possible of the old methods, which still are left using them and conserving them for the future.

In this article the author describes the method of lime water consolidation for old plaster and mural paintings. The author has learned the method in the tradional way and has been using it for consolidation of medieval secco paintings, as well as on later paintings, in Swedish churches. The method is also useful for repair and saving of old plaster and calcareous stone.

To prove such a method scientifically is difficult, because there are no standard measuring techniques to test lime plaster in situ and there isn't any, or very little, litterature. This because in the past craftsmen used only their experience when they tested the quality of a material and seldom wrote. They learned in a practical way, e.g. from father to son.

DESIRABLE PROPERTIES OF A SUITABLE CONSOLIDANT FOR LIME PLASTER, WITH OR WITHOUT DECORATED SURFACES

Before beginning the consolidation of a wall painting, plaster etc.., it is of great importance to be sure that the chosen consolidant and the application method will not do any harm to the object, and that the treatment will not hinder a further consolidation or make this impossible.

When plaster is being consolidated, it has to be strong enough, but not too strong. The consolidant has to penetrate as deep and homogeneously as possible to avoid lamination effect, which will cause stress between the waeker and the stronger layers (Clifton, 1980). These stress points will cause damage in the plaster itself or loosening cohesion from the support, as well as a too strong consolidation of a paint layer always causes flaking or sometimes worse, strappo of the paint.

It is also very important that the chemical composition of the consolidant is as similar as possible to the material which should be consolidated. A new composite material has not to be formed by the consolidant. The consolidant has also to behave similarly in ageing, thermal expansion and shrinking to the original material. Furthermore it should not change the refractive index or cause discolouration or glossiness of the surfaces. A consolidant should not disturb the wall's breathing (Holmström, Sandström, 1972) and it is also important that it does not get acid character by ageing.

Properties of lime water

It is possible to fix and consolidate both the paint layer and the plaster with lime water. The treated material will keep its porosity and the colours will not change.

Properly used, the lime water is a good consolidant, this method was employed to save most of the medieval mural paintings in Sweden (Nisbeth, 1980).

For the consolidation of wall paintings and plaster with calcareous binding media the lime water has the desiderable properties mentioned above. The high pH of lime water causes normally no damage to the plaster or to the mural paintings. Some harms of the method could occur in those parts where the artists were using the tempera technique, e.g. to have the possibility to use a non lime stable pigment and/or to retouch for highlights, etc.... This technique is very common in fresco paintings.

These tempera parts must be protected from the lime water during the consolidation process. If such parts need to be consolidated, it is possible to use calcium bicarbonate solution, which has a lower pH (6). The high pH of lime water has a neutralizing action on acids.

HOW TO MAKE THE CONSOLIDANTS

Lime water

Lime water ($Ca(OH)_2$) is a clear saturated solution, containing 1700 mg Ca/per 1 of H_2O at 20°C, the pH is about 9. In the atmosphere it will form calcium carbonate (Ca CO_3), the reaction is slow.

For the mixture, 1 part of lime putty and 6-8 parts of pure water (proportions by volume) are taken. The mixture must be well stirred and then left to rest for at least 24 hours. After this time the excess of lime will be deposited on the bottom of the vessel with the clear lime water solution over it. On the top of the lime water a hard skin, a crust, is formed. This crust is calcium carbonate, resulting from the reaction between the $Ca(OH)_2$ and the CO_2 of the air.

For the consolidation treatment, the clear lime water must be carefully drawn out from the vessel in order to avoid mixing of the clear solution with the deposit of lime. Before pouring the lime water into a pressure sprayer, used for the consolidation, the new formed crust has to be filtered off.

Calcium bicarbonate

Calcium bicarbonate (Ca CO_3+ H_2O) is a saturated solution, containing 1100 mg Ca CO_3/per 1 of H_2O at 20°C, the pH is about 6. The solution is not stable in contact with the atmosphere, a quick reaction will occur.

To make calcium bicarbonate it is necessary to inject CO_2 gas into lime water kept in a well closed vessel. The transition can be followed by control of the pH, which falls from 9 to 6 (Denninger, 1958).

Calcium bicarbonate could be good for consolidation of tempera parts in paintings, which can be too sensitive to the alkaline action of lime water. Care must be taken when calcium bicarbonate is used, because the rapid reaction will prevent a deep penetration and too strong consolidation of the surface might result.

THE METHOD OF LIME WATER CONSOLIDATION

Consolidation of brittle or friable plaster and mortar, flaking or powdering paint layers in lime technique could be done by spraying with lime water. The spraying is carried out with a pressure sprayer, which gives an even layer on the surface.

Before the consolidation starts, a dry cleaning of dust and loose dirt on the surface must be done. If the surface needs a wet cleaning it could, in many cases, be done by lime water application, which has a caustic character.

The cleaning with lime water is carried out by plentiful spraying the dirty surface and the dirty water is then quickly absorbed from the surface with the help of a natural sponge. It is extremly important not to have flowing water on the surface. A test before the cleaning starts, on a less visible and less important part, should be made.

When the cleaning operation is completed, the consolidation procedure starts by gently spraying on the wall surface with pure tap water to improve the capillary action; the spraying is only done when the wall surface refuses to absorb the lime water solution. In such a way, the pure water, with lower surface tension, will ease the entry of the lime water into the pores and allows further penetration.

When the surface begins to absorb the water, it is time to start the lime water spraying. The treatment goes on until the surface is starting to show glossiness. The excess of consolidant is immediately removed with a sponge, in order to avoid superficial carbonation, which causes efflorescences.

To reach a deep penetration of the consolidation, the spraying is repeated as long as the wall is able to absorb the lime water, 30 - 40 times or even more. The rate of a spraying cycle is depending of the water transport into the wall, which is directly connected with the porosity of the used building material and the physical forces for the water movement inside this material.

After a certain number of spraying cycles the absorption of the lime water by the wall decreases. At this point it is necessary to stop the consolidation, before efflorescences and closing of the pores will occur. Heat, humidity and the presence of carbon dioxide in the air will promote the process of carbonation.

Problems with salts, loosening of plaster from the wall structure, calcium sulphate, etc..., must be solved by special treatments.

FORCES SUPPORTING THE PENETRATION OF THE CONSOLIDANT

Building materials, as stone, brick and mortar, are porous and water moves easy into them, supported by physical forces. These forces act separately or in combination (Torraca, 1981).

Forces as attraction by hydrophilic surfaces, capillary action, electro-osmosis attraction, heat and cold, water diffusion and evaporation could be employed by the restorers. These forces, when well known by the user, could be a valid support to reach a deep and homogeneous penetration.

The characteristics of those forces can be studied in " Porous Building Materials " by Dr. G. Torraca, 1981, where they are well described. Furthermore, collaboration with chemists and scientists is a need for the restorer.

To help the penetration of the lime water, suitable climate should be created, or seasons with proper weather have to be chosen. For example, in winter the vapour pressure is generally higher inside a building wall than outside. It depends on the fact that the indoor air is warmer and can hold more water vapour than the colder outdoor air; it means that the water will move outwards through the wall, except in the walls with vapour-proof barriers. This water flow could be utilized to acquire deep penetration.

Attention should be paid that damage by salts or frost will not occur. Thus, the lime water method should not be used outdoor the last two months before, or during the frost period, in areas where the temperature periodically falls below 0°C, because the risk of frost damage.

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LIME BASED MORTARS FOR THE REPAIR OF ANCIENT MASONRY AND POSSIBLE SUBSTITUTES

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SUMMARY

A better definition of properties to be specified for mortars to be used in the conservation of ancient masonry is necessary in order to prepare the way towards drafting of specifications and testing of materials by standard procedures.

Standard samples of mortars made with lime, lime/cement, lime/pozzolana, lime/brick powder were submitted to the following tests : setting time, compressive strength and modulus, flexural strength, pore size distribution, extraction of alkaline materials.

Results show that it is possible to prepare reliable mortars, easy to apply, with properties not excessively dissimilar from those of traditional materials.

Use of low-alkali cement (or hydraulic lime) would reduce the danger of efflorescence and crystallization damage.

Water-reducing agents, used in modern concrete, are effective also in lime-based mortars.

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1. INTRODUCTION

1.1 General requirements of conservation materials

A general requirement for materials to be used in the repair of ancient structures is that, besides performing satisfactorily in their mechanical function, at the moment of application, they should not cause an acceleration of the deterioration rate of the adjoining, ancient, material. This holds not only for buildings but in general for objects of any type.

Unfortunately this requirement is frequently forgotten in the quest for strong and durable materials aimed at producing long-lasting repairs. Such materials, by themselves, behave quite honourably under environmental stresses but, when bound with ancient weaker, materials they discharge all stresses on them. sometimes even introducing new causes of decay. Actually a new material inserted in old masonry may cause not only mechanical stress (e.g. due to thermal expansion cycles) but also other types of stress of physico-chemical origin (e.g. crystallization of soluble salts formed in the setting processes of mortars, trapping of moisture by non-porous layers).

1.2 Portland cement mortars in conservation

A typical example of failure to consider the future effect of a "strong" modern material in a comparatively "weak" context is constituted by the use of Portland Cement mortars and of concrete elements in the repair of traditional brick and stone masonry.

From the point of view of the general principle discussed in the previous paragraphs Portland Cement has the following defects :

(a) Portland Cement is too strong (high compressive strength and high modulus). In case of differential movement, stress will be transmitted to the older sections which will fail.

(b) Portland Cement mortar and concrete have a large thermal expansion coefficient (which may even be twice as large as that of lime mortars and of most types of brick and stone). Thermal cycles result therefore in stress and damage of the "weak" old masonry.

(c) Portland Cement forms soluble salts while setting and leaching of soluble salts takes place even a long time after setting when mortar or concrete come in contact with rain-water.

Soluble salt solutions may be transmitted into nearby porous materials causing damage by crystallization when the water evaporates.

(d) Portland Cement mortars have a low porosity largely consisting of very small pores - layers of cement mortars hinder water movement in the masonry. This might look beneficial at first sight (e.g. inhibition of penetration of rainwater) but in most cases it ends up by causing damage because of accumulation of moisture behind cement layers that do not allow evaporation.

1.3 Lime mortars in conservation

As the large use of cement in the conservation of ancient structures in the last 80 years resulted in quite a number of examples of the defects listed above, the modern trend in recent times has been to restrain its use.

Since most ancient masonry in Europe is made of lime-sand mortars, there has been a tendency to specify the use of traditional lime mortar for masonry repair. This, in turn, has met with other difficulties.

(a) The use of lime mortar requires considerable skill on the part of the mason. Water addition improves workability but tends to reduce mechanical strength. A good balance is not easy to find.

(b) Lime mortars are slow-setting and require a relatively dry environment. In ancient masonry, which is frequently moist, lime may set very poorly or not at all.

(c) Setting of lime depends upon carbon dioxide from the air reacting with the mortar. In thick structures, carbonation of the core is extremely slow and unreliable.

From the mechanical point of view, the properties of well-set lime mortars are quite adequate. Furthermore they present some peculiar characteristics that may be regarded as desirable.

In particular lime-mortars show a relatively high deformability (low modulus), which allows them to accommodate some differential movement, and a good proportion of large pores in their pore-size distribution which should favour moisture transmission and evaporation.

1.4 Lime-cement mortars

In an attempt to combine the hydraulic properties and ease of use of cement with the low strength, high deformability and porosity of lime, mixed lime-cement mortars have been brought into use and have gained considerable acceptance (BRE, 1973).

As has already been noted, (Holmstrom, 1977), even this combination is not exempt from defects. In the first place, if the mixed mortar sets under relatively dry conditions it is definitely worse than a pure lime mortar as to mechanical properties, because the cement fraction does not set immediately and acts as an inert filler. Vice-versa in moist conditions lime would not set (or set only in part) while cement would. It is obvious, therefore, that the mechanical properties obtained in any real situation will not be exactly predictable and may be rather poor in a dry environment.

Secondly, if the cement proportion is rather high, the amount of soluble salts formed will also be high, even if lower than in the case of pure cement.

1.5 Hydraulic additions to lime mortars

Lime mortars may be made to set under moist conditions if pozzolanic materials are added to the mixture.

Such materials include pozzolana proper, fly ash and trass.

They have been used since a very long time and their use has been frequently successful.

Besides the fact, however, that these materials also form soluble salts in the setting reactions (even if not the same ones as Portland Cement), the mechanical properties of lime-pozzolana mixtures have not been studied at all and the right proportions to obtain mortars suitable for conservation are far from being established.

Also the addition of crushed ceramic bricks or tiles introduces definite hydraulic properties in lime mortars. Such mortars appear to be weaker than the pozzolanic ones but the amount of soluble salts they may contain should be far smaller.

Also for the materials in this class the same limitation applies as for the addition of cement to lime : if the setting takes place in a dry environment, mechanical properties are worse than those of lime alone.

1.6 The ideal conservation mortar and possible improvement of the existing ones

As the mechanical properties of lime based mortars and of traditional masonry are very imperfectly known, it is, at the present moment, quite difficult to set down the detailed requirements applicable to a mortar for masonry restoration. As things are now, some general principles may be stated tentatively.

(a) A restoration mortar should have values of mechanical strength and modulus in the same range as those of a good lime-sand mortar.

(b) Pore-size distribution should be comparable with that of a lime-sand mortar.

(c) The mortar should contain the smallest possible amount of soluble salts (in particular of sodium and potassium salts).

(d) The mortar should be easily workable (without requiring highly specialized technical skill).

(e) The mortar should set, with sufficient rapidity and reliability both in a dry and in a wet environment.

Should the last requirement prove too difficult to fulfill by any one material (suitable for both wet and dry conditions) one might consider the possibility of specifying different mortars according to the humidity present in the environment (air or old masonry).

1.7 Testing of mortars for conservation

It is obvious that mortar mixes should be submitted to standard tests in order to ascertain their compliance to the requirements of conservation mortars.

Unfortunately the available test standards are far from adequate.

Tests for mortars have been frequently modelled on cement mortars; consequently they require substantial modification in order to be applicable to lime-based mortars (Davison, 1976).

Definition of the properties to be measured constitutes already a problem in itself and the following discussion is meant to be only an initial, tentative statement.

(a) Quality control of materials

Specifications for lime, sand, cement and pozzolanic materials do exist but are likely to need some revision to be adapted to working conditions in restoration projects. Attention should be given to hydrated lime putty in order to allow easier execution of quality control tests.

A preliminary attempt (two measurements only) showed that thermogravimetric analysis might be a useful test as it allows the proportions of calcium hydroxide, calcium carbonate and water in the putty to be established rapidly. It should also indicate the presence of magnesium hydroxide (as it de-hydrates at a different temperature to that of calcium hydroxide) but it was not possible to ascertain such a fact in our case.

(b) Workability

A complex property which could possibly require a combination of tests to be measured in a realistic way (Davison, 1976).

Apparently none of the tests currently available yield results which correlate well with the opinion of a competent mason, although several of them are in current use (ASTM C 780-74, cone penetrometer; BS 4551, 1970, flow table and dropping ball).

According to Davison assessment of the response of the mortar to the trowel by a mason remains still the most reliable definition of workability.

(c) Setting time

Application of available standard techniques should pose no problem.

(d) Volume change upon setting

Some water mixes contract more than other ones upon setting. Such data should be quantified by measurements carried out on samples setting under controlled conditions.

(e) Mechanical properties

Testing of flexural and compressive strength, according to current specifications, should be adequate. When new types of mortars are tested the measurement of modulus might be required.

Mixing and curing conditions specified in the standards should be adapted to the requirements of the various lime mixtures. In particular the amount of water added should be exactly defined, since the mechanical strength of the hardened sample depends strongly upon it. Tests under "dry" and "wet" curing conditions should also be specified.

(f) Adhesion to masonry units (brick or stone)

Adhesion is an important property as it determines the resistance of the masonry to some types of stresses and the good behaviour of joints against water penetration.

Adhesion, however, is seldom measured, even if specifications exist (ASTM E 149-66), because preparation of samples is costly while results are very dispersed (so that large numbers of samples should be tested to obtain significant data).

As such difficulties make adhesion testing unattractive, one wonders whether it could be substituted by testing the overall performance of assembled masonry units under stress or against water penetration.

(g) Thermal expansion coefficient

An important measurement, particularly for mortars to be used in concretes for casting reinforcing elements. It should be required for testing new types of mortars but not as a routine quality control test.

(h) Soluble salts

An essential test for mortars used in conservation as it allows the assessment of potential danger to original elements of masonry which come in contact with the new mortar.

It could be carried out either by analysis of alkaline elements in the binder (e.g. by atomic absorption spectrometry) or by extraction with cold or hot water of hardened mortars.

A direct way of measuring the degree of danger is constituted by efflorescence tests (Ritchie, 1955) which, however, are difficult to quantify.

A specification for binders for conservation mortars should contain a maximum allowable limit of alkaline elements (or soluble salts).

Simple quality control tests, usable on worksites, should also be developed.

(i) Weathering resistance

As the most important mechanism of fracture of mortars in masonry is salt crystallization, the inherent resistance of a mortar to decay should be tested by salt crystallization tests, as applied to stone (e.g. UNESCO RILEM, 1978).

Unfortunately, the test is based on the use of Sodium Sulphate as the crystallizing salt, and mortars containing Calcium Carbonate and Aluminium compounds undergo a particularly rapid deterioration in the presence of Sulphates (due to the formation of Calcium Aluminium Sulphate, Ettringite) resulting frequently in the destruction of samples in one cycle.

The test, as it is, is inapplicable and a new one should be devised; one wonders, however, whether this is necessary at all.

In the first place, according to the general requirements stated in paragraph 1.1, the restoration mortar should not be required to have a weathering resistance far superior to that of the original mortar. In particular, it should not be water repellent, but should absorb its generous share of the water circulating in the masonry pores.

When it comes to crystallization of soluble salts, and fracture, there is nothing wrong if the material is able to withstand it for a while without excessive damage; this is determined mainly by its tensile strength, which, in turn, is reflected in the flexural strength measurement.

A conservation mortar should therefore have a compressive strength not much in excess of that of the original mortar, but, if possible, a flexural strength not excessively below the value of its own compressive strength. It should also have a tendency to absorb water comparable to that of the original mortar.

While this set of properties might adequately define the inherent weathering behaviour of the material, the salt crystallization test would be inadequate, and even dangerous, because it would offer an advantage to low porosity or water repellent materials which tend to discharge the solutions in contact with the masonry onto the original parts and so to accelerate their decay.

(j) Water absorption and porosity

In view of what has been said about weathering in the preceding paragraph, the determination of the pore structure and water absorption of a mortar is a rather important one.

Adequate methods exist for the determination of water absorption and porosity (UNESCO/RILEM, 1978).

Pore size distribution should be determined (with a mercury porosimeter) as the proportion of large to small pores plays an important role in the weathering processes and in the distribution of water between adjacent materials. It must be kept in mind, however, that such expensive laboratory procedures are applicable to the study of new untested mixtures, and not to continuous quality control on work site.

2. RESEARCH PROGRAMME AND ORGANIZATION

2.1 Programme

The ICCROM research programme started in 1979 and ended in 1981. The main goal of the project was to study lime, lime-cement, and lime-pozzolana mortars in order to quantify, as much as possible, mechanical and physico-chemical characteristics relevant to conservation problems, according to the lines of thought developed in the previous paragraphs.

Samples of mortar were to be prepared, according to existing specifications, whenever available. Samples were to be tested for : flexural strength, compressive strength, modulus under compression, alkaline elements concentration, total porosity and pore-size distribution.

In the second year of work also a lime-crushed brick mortar and measurements of setting time, depth of carbonation and water absorption were added to the programme.

Salt crystallization tests and efflorescence experiments were attempted but abandoned because of the limited success of the experiments.

It was decided to prepare a fairly large number of samples for each test in order to be able to obtain more reliable figures. This, in turn, resulted in the requirement of more samples per mixture and caused a reduction of the number of formulas that could be considered for testing.

The second goal of the project was to explore possible modifications of the (more or less) traditional mortars studied in the first group of tests. The following ideas were chosen for preliminary testing :

(a) Application of water reducing admixtures to lime-based mortars.

(b) Use of low-alkali cement in lime-cement mixtures.

(c) Use of fillers in Portland cement to reduce mechanical strength and increase porosity.

(d) Use of hydraulic limes as binders.

A small number of samples was prepared for these experimental mortars with the aim of opening new research lines rather than that of defining properties in a reliable way.

2.2 Organization

Fellowships provided by EEC (Economic European Communities) were used to constitute two teams each composed of two architects from the Scuola di Perfezionamento per lo Studio ed il Restauro dei Monumenti of Rome University and one engineer from the Istituto di Scienze delle Costruzioni of the Faculty of Engineering of Rome University.

One group was active in 1979 and the second in 1980/81.

Both groups were assisted by experts from ICCROM and C.N.R. (Centro di Studio Cause di Deperimento e Metodi di Conservazione delle Opere d'Arte, Rome).

Samples were prepared in the ICCROM laboratory where simpler tests could also be performed.

Mechanical measurements were carried out at the Istituto di Scienze delle Costruzioni. Chemical analysis and porosity measurements at the CNR laboratory.

The research programme had in part a training value for the young architects and engineers involved in the groups (which were denominated, for that reason, Research Training Units). It had been planned that one of the aims would be that of introducing young specialists to the problems of architectural conservation.

This double aspect of the project (research and training) should be kept in mind when evaluating the results obtained in the programme and may help explain some inconsistencies in its development.

3. PREPARATION OF MORTARS

Mortars were prepared by gauging, mixing, moulding and curing under the general climatic conditions indicated by Italian specifications (DM, 1968) i.e. 20 $\pm$ 2 C and 75 $\pm$ 5% relative humidity.

Such conditions were maintained in an isolated room by means of a thermo-humidifier (plus the central heating system of the house) and monitored by a hair thermo-hygrograph.

3.1 Gauging

Detailed data on the materials used to make the mortars are listed in Annex 1.

In the first year of the project hydrated lime powder was used for the lime-based mortars. In that period proportions of all components were gauged by weight.

In the second part of the project lime putty was used and all proportions had to be gauged by volume.

In the tables of results all proportions are now indicated by volume.

In the development of the research programme, several mortars were prepared and proportions were often varied in an attempt to improve the performance.

In order to designate the various compositions in a simple way the following symbols were adopted :

C	=	Portland Cement
L	=	Lime putty
L^+	=	Hydrated lime powder
Lh	=	Hydraulic lime (Italcementi)
$L^{++}h$	=	Hydraulic lime (Lafarge)
Cla	=	Cement, low-alkali
S	=	Sand
P	=	Pozzolana
B	=	Brick powder
X	=	Calcium carbonate
M	=	Marble powder

Volume proportions are indicated by subscript numbers.

When water-reducing agents are used these are also indicated by symbols, in the mortar formula, without subscript, as the proportions used are small.

Rb	=	Rheobuild 561 (liquid) 8 - 11 cc per Kg. of binder
Fl	=	Fluid Standard (liquid) 8 - 16 cc per Kg. of binder
Gl	=	Sodium Gluconate (solid) 4 cc of 30% solution per Kg. of binder

The amount of water required to achieve good workability was assessed for each mortar in a practical way, i.e. by consultation with a mason working on a restoration programme inside the San Michele building where ICCROM is located.

3.2 Mixing

A mixer designed to Italian specifications (DM, 1968) was used.

It operates at two speeds : 140 ± 5 rpm and 285 ± 10 rpm.

DM 1968 specifies mixing times, at both speeds, for cement and hydraulic lime (2 minutes and 30 seconds, respectively) and these were applied in our case. For the other mortars, mixing times were modified by us in order to achieve the best possible homogenization of binder and aggregate keeping in mind the prescriptions of the ACI Building Code (ACI, 1978).

Mixture (Order of addition)	Time at 140 ± 5 rpm	Time at 285 ± 10 rpm
H_2O, C, S	1'	1' 30"
H_2O, Lh, S	1'	1' 30"
L^+, S, H_2O	2' 30"	2' 30"
L, S, H_2O	6'	4'
L, S, H_2O, C (CPa)	2' 30"	1'
P, S, L, H_2O	5'	2'
B, S, L, H_2O	5'	4'
S, X, H_2O, C	9'	3'
M, X, H_2O, C	9'	3'

When water-reducing agents were used they were added at the end and mixed one minute at each speed.

3.3 Moulding

Stainless steel moulds, holding three pieces (4 x 4 x 16 cm.) each, designed according to the Italian (DM, 1968) specification were used to mould specimens for mechanical testing.

Chemical and physical tests were carried out on cubic samples (5 x 5 x 5 cm.) moulded in PVC moulds.

Both types of samples were compacted according to the procedure prescribed by the Italian specification (DM, 1968) which is equivalent to the ISO specifications.

The compacting machine drops the mould at regular intervals. Initially the moulds are filled half way up and mortar is slightly compressed into them by hand; this first fill is then made compact by 60 strokes of the machine. Afterwards the mould is completely filled and another series of 60 strokes is applied.

In the case of all lime-based mortars two layers of filter paper are placed at the bottom of the mould, before filling, to simulate the sucking action of bricks.

3.4 Curing

Cement-sand mortars were submitted to the conditions prescribed by the Italian specifications (DM, 1968); that is 27 days of immersion in water.

Such conditions, obviously, could not be applied to Lime-sand mortars which require air drying; they had also to be modified for mixed mortars as these set more slowly than the Cement-sand mixtures.

For ease of presentation of the result in the tables, symbols were adopted also to designate the curing cycles used, as explained here below :

Symbol	Total Time (days)	Conditions	Temperature
D_1	60	at 75% RH	20 ± 2%
D_2	60	4d at 90% - 56d at 75% RH	"
D_3	60	7d at 90% - 53d at 75% RH	"
M	28	at 90% RH	"
W_1	28	1d at 90% RH - 27 d in liq. H_2O	"
W_2	28	4d at 90% RH - 24 d in liq. H_2O	"
W_3	28	3d at 90%+ - 4d at 90% RH - 21 d in liq. H_2O	"

+ Note : Samples covered with a polyethylene film

4. TESTING METHODS

4.1 Setting time

The Vicat needle was applied according to ASTM C 191 - 77.

The mortar sample is poured into a special truncated-cone mould. Initial setting time is taken as corresponding to a penetration of 25 mm of the plunger.

4.2 Mechanical strength and modulus

Flexural strength was determined, at the end of the curing cycle, on the prismatic specimens. The Italian testing specification, which was followed, is equivalent to ASTM C 109 or C348 (and several European ones).

Compressive strength is determined on both ends of the prism broken in the flexural test.

For the measurement of modulus, the (4 x 4 x 16 cm.) prismatic specimens were compressed lengthwise while two strain gauges were applied on two parallel faces. At loading intervals of 5 Kg., the strain was recorded and the stress-strain relationship was plotted.

The graph allows a tangential modulus to be calculated, from the initial section of the curve, and a final (or secant) modulus, from the value of strain at the breaking point.

The difference between the two values offers an indication of the extent of irreversible deformation that takes place before fracture.

4.3 Soluble alkaline substances

Cubic samples (5 x 5 x 5 cm.) of mortar were crushed, powdered, separated in three fractions by sieving (1 - 2 mm, 0.5 - 1 mm, 0.5 mm). 10 g of each fraction were weighed and combined in an Erlenmeyer flask.

In each flask 250 ml of bi-distilled water were added and the flasks were closed. The flasks were shaken manually once a week.

After 30 days the supernatant liquid was filtered off in a 500 ml. volumetric flask and the residual solid was washed carefully with bi-distilled water that was also filtered off in the volumetric flask.

The solution was then brought up to volume by a last addition of bi-distilled water; aliquots were then analyzed for Sodium and Potassium by means of an absorption spectrophotometer (Model 603, Perkin-Elmer).

Results are expressed in equivalent weights (or rather milli-equivalents) per kilogram of mortar, in order to allow easy comparison of different materials and an overall evaluation of the degree of dangerousness.

In the mortars, made of standard Italian sand, however, alkaline material may also be provided by the sand, through ion exchange between calcium ions in solution and sodium or potassium ions in the plagioclase.

This is clearly shown by the small, but sizable, amount of alkaline material produced by Lime-sand mortars (some of this could also be contained in the lime).

4.4 Pore size distribution

Measurements were carried out on small irregular blocks, carved out of the 5 x 5 x 5 cm. cubes, using a mercury porosimeter (Type 1500, Carlo Erba).

The technique of measurement and the way to express results were recently standardized by an Italian Committee concerned with tests for stone conservation (Normal F, 4/80).

4.5 Water absorption

Total water absorption was measured according to a recent Normal specification (Normal F 27/80).

This specification prescribes the measurement of weight increase in immersed specimens at regular intervals and allows also the measurement of absorption rates.

Water absorption curves obtained showed, however, that most of the lime-based mortars absorbed water up to a saturation level within the first five minutes, which is the first measured point in the absorption curve. The only exception is constituted by a $L_4 C_1 S_{10}$ lime-cement mortar, with a low water/cement ratio (0.69) which showed a slow water absorption (comparable to that of stone or brick).

4.6 Other tests

Other test methods were tried in the course of the research programme but their results are not shown in full in the present report as they did not appear sufficiently indicative or reproducible.

The carbonation process of the lime-based mortars was investigated by applying a 1% hydroalcoholic solution of phenolphtalein on the freshly broken surfaces of a sample at the end of the curing cycle, as suggested by BRE IS 14, 1978 (Fresh surfaces where calcium hydrate is still present are coloured purple).

The phenolphtalein test showed that in most lime-based mortars only a superficial external carbonation of a few millimetres occurred after the standard curing period. For pure lime mortar and for a lime-pozzolana mortar ($L_4\ P_1\ S_{10}$) the carbonation process appeared to be just started. In the case of hydraulic mortars with addition of calcium carbonate it was noticed that good carbonation had taken place also inside the sample. The X-ray diffraction carried out by S. Z. Lewin on several samples after six months (private communication of work still unpublished) showed that almost complete carbonation had been achieved.

Evaluation of efflorescences produced on brick by mortars was attempted on a special set of cubic samples in which a terracotta tile is unfixed (Ritchie, 1955). Two samples were prepared for each type of mortar. Results appeared to be poorly reproducible, both from the qualitative and the quantitative point of view. Such difficulties might be connected to the presence of calcium bicarbonate in the liquid phase or to the imperfect adhesion between mortar and tile.

5. RESULTS

Results of mechanical, physical and chemical tests are reported in Tables I, II and III.

The data available allows some tentative conclusions of a qualitative character to be drawn on the properties that are most interesting in relation to the conservation of brick or stone masonry.

L = lime putty   L* = hydrated lime powder

TABLE Ia Water/Binder ratio, Curing, Setting time and Mechanical properties

Mortars [x] proportions by volume	Water/Binder ratio°°	Curing cycles		Initial setting time (days)	Mechanical properties			
		Conditions°	Time (days)		Flexural strength MPa	Compressive strength MPa	Elastic modulus MPa x 10^3	final E
$C_1 S_{2.3}$	0.50	W_1	28	1/6^	6.41±0.36 (15)	38.9±2.64 (30)	30 - 12^	19 - 10^
$L^*_1 S_1$	1.00	D_2	60	n d	0.43±0.07 (9)	0.50±0.07 (18)	n d	n d
$L^*_1 S_{2.6}$	1.00	D_2	60	n d	0.58±0.12 (11)	0.59±0.18 (22)	0.75±0.16 (2)	0.25±0.01 (2)
$L_1 S_3$	1.72	D_1	60	8	0.62±0.05 (12)	0.91±0.13 (24)	5.56±0.71 (6)	4.23±0.42 (6)
$L^*_4 C_1 S_6$	0.72	M	28	n d	0.90±0.12 (17)	2.82±0.59 (34)	n d	n d
$L_4 C_1 S_{10}$	0.69	D_3	60	1	0.89±0.08 (19)	2.70±0.73 (38)	5.09±0.01 (2)	2.96±0.05 (2)
$L^*_4 C_1 S_{16}$	0.99	M	28	n d	0.49±0.98 (15)	0.86±0.23 (30)	0.51±0.19 (3)	0.19±0.09 (3)
$L_3 C_1 S_{12}$	1.29	D_3	60	3	0.62±0.06 (12)	0.91±0.13 (24)	1.04±0.07 (6)	0.54±0.16 (6)
$L_3 C_1 S_{12}$	1.29	M	28	n d	0.51±0.08 (4)	2.17±0.11 (8)	3.48±0.06 (2)	1.13±0.01 (2)
$L^*_1 P_{1.2} S_{2.5}$	1.30	W_2	28	n d	2.11±0.86 (15)	9.01±1.22 (30)	0.92±0.22 (3)	0.47±0.04 (3)
$L^*_1 P_{1.2} S_{2.5}$	1.30	D_2	60	n d	1.44±0.73 (18)	5.58±2.08 (36)	1.95±0.43 (2)	0.74±0.23 (2)
$L_4 P_1 S_{10}$	1.38	D_3	60	8	0.14±0.05 (12)	0.77±0.15 (24)	1.04±0.29 (6)	0.58±1.62 (6)
$L_4 P_1 S_{10}$	1.38	M	28	n d	0.19±0.03 (4)	0.73±0.21 (8)	1.01 (1)	0.58 (1)
$L_1 B_1 S_2$	1.76	D_3	60	10	0.25±0.03 (12)	0.75±0.05 (24)	1.52±0.08 (6)	0.96±1.27 (6)

Note: [x] For key to component symbols in mortar composition see paragraph 3.1; °° Water contained in lime putty (approx. 50%) was enclosed in the calculation; ° For key to curing conditions symbols see paragraph 3.4; ^ Data obtained from the literature; () The number of measurements is indicated in brackets after each average result.

TABLE Ib Water/Binder ratio, Curing, Setting time and Mechanical properties

Mortars* proportions by volume	Water/Binder ratio°°	Curing cycles		Initial setting time (days)	Mechanical properties			
		Conditions°	Time (days)		Flexural strength MPa	Compressive strength MPa	Elastic modulus MPa x 10^3 tangential E	Elastic modulus MPa x 10^3 final E
$L_3\ Cla_1\ S_{12}$	1.29	D_3	60	3	0.50±0.05 (4)	2.20±0.17 (8)	3.64±0.24 (2)	2.67±0.31 (2)
$L_3\ Cla_1\ S_{12}$	1.29	M	28	n d	0.33±0.05 (4)	1.09±0.05 (8)	2.22±0.01 (2)	1.42±0.01 (2)
$Lh^{\leftrightarrow}_1\ S_{1.6}$	0.40	W_3	28	n d	2.71±0.50 (8)	7.82±2.94 (16)	n d	n d
$Lh_1\ S_{1.6}$	0.55	W_3	28	1/2	2.02±0.05 (4)	7.46±3.73 (8)	9.11±0.86 (2)	6.66±1.18 (2)
$C_1\ X_2\ S_9$	2.46	M	28	n d	0.64±0.01 (2)	2.74±0.02 (4)	4.51 (1)	1.87 (1)
$C_1\ X_2\ M_9$	2.46	M	28	n d	0.61±0.02 (4)	2.32±0.21 (8)	2.89±0.16 (2)	1.01±0.23 (2)

TABLE Ic Water/Binder ratio, Curing, Setting time and Mechanical properties

Mortars[x] proportions by volume	Water/Binder ratio°°	Curing cycles		Initial setting time (days)	Mechanical properties			
		Conditions°	Time (days)		Flexural strength MPa	Compressive strength MPa	Elastic modulus MPa x 10^3 tangential E	final E
$L_1 S_3$ Rb	1.28	D_1	60	7	0.93±0.05 (4)	2.10±0.11 (8)	8.05±0.01 (2)	4.71±1.18 (2)
$L_1 S_3$ Fl	1.36	D_1	60	n d	0.88±0.08 (4)	1.96±0.11 (8)	3.48 (1)	2.88 (1)
$L_3 C_1 S_{12}$ Gl	0.98	D_3	60	n d	0.81±0.08 (4)	3.90±0.16 (8)	5.09 (1)	4.05 (1)
$L_3 C_1 S_{12}$ Rb	0.98	D_3	60	2	0.84±0.06 (12)	4.15±0.48 (24)	7.62±0.59 (6)	4.38±0.33 (6)
$L_3 C_1 S_{12}$ Fl	1.05	D_3	60	n d	0.67±0.07 (6)	3.13±0.55 (12)	4.55±0.43 (3)	2.53±0.69 (3)
$L_4 P_1 S_{10}$ Rb	1.19	D_3	60	8	0.61±0.01 (2)	1.11±0.11 (4)	2.78 (1)	2.40 (1)
$L_4 P_1 S_{10}$ Fl	1.23	D_3	60	n d	0.45±0.07 (4)	0.91±0.08 (8)	2.91 (1)	2.82 (1)
$L_1 B_1 S_2$ Rb	1.45	D_3	60	10	0.73±0.04 (4)	1.53±0.12 (8)	3.36±0.66 (2)	2.31±0.20 (2)
$L_1 B_1 S_2$ Fl	1.45	D_3	60	n d	0.57±0.03 (4)	1.79±0.04 (8)	3.48±0.11 (2)	2.55±0.07 (2)
$L_3 Cla_1 S_{12}$Rb	0.98	D_3	60	1/2	0.67±0.06 (4)	4.27±0.05 (8)	5.21±0.50 (2)	3.43±0.27 (2)
$L_3 Cla_1 S_{12}$Fl	0.98	D_3	60	n d	0.68±0.08 (4)	4.32±0.09 (8)	5.23±0.18 (2)	3.67±0.15 (2)

TABLE II Pore size distribution in mortars

Mortars	V% Percent of total pore volume (pore diameter in µm)												
	ρa (g/cm^3)	P%	d < 0.05	d 0.05-0.1	d 0.1-0.2	d 0.2-0.4	d 0.4-0.6	d 0.6-0.8	d 0.8-1	d 1-2	d 2-4	d 4-10	d > 10
$C_1\ S_{2.3}$ (3)	2.15±0.02	16±1	47	19	19	5	4	3	1	2	0	0	0
$L_1^*\ S_{2.6}$ (3)	1.90±0.02	25±2	22	5	8	13	6	3	3	3	2	5	30
$L_1\ S_3$ (4)	2.14±0.02	25±1	14	5	13	12	1	1	1	3	2	3	45
$L_4^*\ C_1\ S_6$ (6)	1.85±0.02	30±1	20	8	9	13	11	8	6	14	4	2	3
$L_4\ C_1\ S_{10}$ (4)	2.10±0.02	22±1	18	12	22	27	9	4	2	6	0	0	0
$L_3\ C_1\ S_{12}$ (4)	1.99±0.02	28±1	15	6	8	10	13	17	8	9	6	3	5
$L_1\ P_{1.2}\ S_{2.5}$ (3)	1.90±0.02	32±1	23	6	7	15	11	6	5	9	7	7	4
$L_4\ P_1\ S_{10}$ (4)	1.93±0.02	30±1	12	5	7	17	10	4	2	3	3	2	35
$L_1\ B_1\ S_2$ (4)	1.90±0.02	36±1	15	5	5	12	8	5	2	6	4	3	35

Note For Water/Binder ratio and Curing cycles of mortars see Table I; () The number of measurements is indicated in brackets after each mortar composition.
ρa = Bulk density; P = Integral open porosity

TABLE III Soluble alkaline material in mortars

Mortars	Sodium (Na)meq/Kg	Potassium (K)meq/Kg	$\Sigma Na^+ + K^+$ meq/Kg
$C_1, S_{2.3}$	8.99	20.72	29.71
$L_1^*, S_{2.6}$	2.35	1.37	3.72
L_1, S_3	2.96	1.29	4.25
L_4^*, C_1, S_6	3.62	9.46	13.08
L_4, C_1, S_{10}	3.47	3.97	7.44
L_3, C_1, S_{12}	3.40	7.45	10.85
$L_1^*, P_{1.2}, S_{2.5}$	5.79	6.48	12.27
L_4, P_1, S_{10}	3.03	7.35	10.38
L_1, B_1, S_2	1.80	4.85	6.65
L_3, C_1, S_{12} Rb	3.76	6.29	10.05
L_3, C_1, S_{12} Fl	1.51	6.43	7.94
L_3, Cla_1, S_{12}	2.74	3.69	6.43
$Lh_1, S_{1.6}$	1.95	5.36	7.31
C_1, X_2, S_9	5.21	14.99	20.20

Note All results reported are the average of two measurements

Our comments will be presented by class of material.

5.1 Cement/sand

In the samples examined cement confirmed its high strength, with flexural strength about one sixth of the compressive one. 66% of the pores were smaller than 0.1 micron diameter and no pore was above the 2 micron diameter.

The amount of alkaline elements extracted in cold water was over seven times that extracted from a lime mortar.

5.2 Lime/sand

Samples of pure lime mortar confirmed some well known peculiarities but offered also some data which was rather surprising.

Strength was much lower than that of cement mortar, but flexural strength was quite close to compressive strength (This might be due to incomplete carbonation after 60 days).

In general mortars made from hydrated lime powder were a little weaker than those from lime putty. The big discrepancy on modulus values between putty and powder may be due to some special reason (Like inferior lime-sand adhesion; although this should influence also flexural strength which, to the contrary, does not appear to undergo a comparable change).

In the "high modulus" lime mortar samples the closeness between the values of the tangential and secant modulus shows that it is a very rigid material not allowing much "plastic" deformation.

The "low modulus" lime mortars show instead a larger deviation from proportionality. Such non-linear, irreversible deformation may be hardly termed "plastic" and is mostly attributed to microscopic cracking in the sample (before macroscopic failure); we venture to suggest that the shape of the stress-strain curve (and the modulus values) must be strongly influenced by defects in the moulding process (including poor adhesion of lime on sand, as shown by SEM pictures taken by S.Z. Lewin, as yet unpublished).

The "plasticity" of lime mortar appears therefore as the result of imperfections in the casting of some mortar pieces rather than as an inherent property of the material.

As such imperfections would impair the resistance to weathering, this presumed plasticity should be considered as a defect rather than a favourable asset (unless one thinks that anything that allows masonry to fail in the joints, rather than in the brickwork, is a favourable factor).

The considerable fraction of large pores is a characteristic of lime mortar (35 - 48% above 10 microns) and the low amount of extracted alkaline elements (about 4 meq/kg.) which may in part originate from the sand.

The slow setting of lime was also confirmed in our test (initial setting time : 7 days).

Improvement of mechanical properties by water-reducing admixtures is discussed in section 5.10.

5.3 Lime/Portland Cement/Sand

Some mixtures behaved quite honourably even in rather dry curing conditions (e.g. $L_4 C_1 S_{10}$ and $L_3 C_1 S_{12}$) showing a definite increase in compressive strength and a slight one in flexural strength (with respect to pure lime mortar).

Modulus showed the same inconsistency of results discussed in the previous paragraph.

Porosity below 0.1 microns compared well with that of lime, but the large porosity (above 10 microns) disappeared altogether. This may influence permeability to water vapour, but in what proportion it is not possible to say.

Mechanical properties obtained with hydrated lime powder were not worse than those shown by cement/lime putty samples (one exception is the high modulus of one putty sample, $L_4C_1S_{10}$, to which, however, no further water at all was added in the mixing stage).

Extracted alkaline elements ranged between 2 and 2½ times the amount extracted from lime mortar. Two setting time determinations were not in agreement with each other, but rather low anyway (1 - 3 days).

5.4 Lime/pozzolana/sand

Two series of samples were prepared with a large difference in pozzolana content.

The "high pozzolana" series yielded strong samples in both dry and wet curing cycles, but the extracted alkaline elements were about three times those of pure lime samples.

The "low pozzolana" series showed an insufficient flexural strength (in a dry curing cycle) while extracted alkalis were still 2½ times those of lime mortar.

In the "low pozzolana" series the fraction of large pores (35% above 10 microns) was preserved and the setting time was as long as that of lime mortar (8 days).

5.5 Lime/brick powder/sand

Flexural strength was slightly higher than that of "low pozzolana" samples, but still insufficient.

The extracted alkaline elements were only 50% above those from pure lime.

The high percentage of large pores, typical of lime mortar, was also preserved (35% above 10 microns).

Improvement by water-reducing admixtures brings these mortars into a useful range of mechanical properties (See section 5.10) but they are unfortunately very slow in setting (10 days setting time).

5.6 Lime/low alkali cement/sand

The formula $L_3 \, Cla_1 \, S_{12}$ yielded samples with sufficient mechanical strength, even in a dry curing cycle.

The low content of extractable alkaline elements was confirmed by the analysis ($1\frac{1}{2}$ the amount extracted from lime mortar, the lowest result among the modified lime mortar).

Setting time was sufficiently fast (3 days) but definitely not very fast.

Pore size distribution was not measured.

5.7 Hydraulic lime

Setting was fast and mechanical properties definitely strong (a little too strong). Alkaline elements extraction gave a not too bad result (less than twice the amount extracted from lime-mortar).

Strength increased when less water was added in mixing.

Pore size distribution was not measured.

5.8 Calcium carbonate as a filler

A satisfactory reduction of the mechanical properties of cement mortars was obtained but, unfortunately, it appears that the alkali content of the chemically precipitated carbonate is sufficient to cause trouble (almost four times as many alkaline elements as from lime!).

Use of marble powder in the place of sand aimed to obtain a white mortar and in fact achieved a satisfactory mechanical behaviour. Another source of fine calcium carbonate powder will have to be used however (fine marble dust?) to solve the problem of alkaline substances.

5.9 Water-reducing admixtures

A consistent result in our studies is that water-reducing admixtures of any type modify mechanical properties of lime-based mortars in a spectacular way.

In pure lime mortar, compressive strength is increased by about 100% and flexural strength by about 50%. Compressive strength increase is even larger in lime/cement mixtures, but the difference between compressive and flexural strength values is much larger than in lime mortar.

Modulus results are more consistent and, on average, higher showing that better adhesion of binder to sand might be a factor in the performance of the admixtures. In the case of the $L_3 C_1 S_{12}$ the increase in compressive strength was over 350%, in flexural strength about 10% and in modulus almost 700% (!).

The "low-pozzolana" mortar showed an improvement but remained slightly below a good lime mortar insofar as mechanical properties are concerned.

Admixtures based on naphto-sulphonates (Rheobuild 561) do not appear to influence the extraction of alkaline elements. Sodium gluconate would add about 1 meg/kg. of sodium (at the proportions used) but its use is not advisable anyway as it slows down the setting reaction of cement.

The effect of water-reducing admixtures on porosity should be a sizeable one, however, as the amount of water added for mixing is sharply reduced (although this might be compensated, in part, by air-entraining).

Unfortunately, these experiments being the last of the series, measurements of pore size distribution could not be carried out in time.

6. CONCLUSIONS

6.1 Criteria for selection of mortars

One aim of the present research project was to favour a process of standardization of the mortars used in the conservation of historic buildings.

We are still rather far from the objective of drafting specifications for each particular application (e.g. repointing, repair, rendering) the main reason being that testing procedures still require substantial improvement.

For instance, a specification for the mechanical testing of lime-based mortars should contain detailed instructions on mixing, casting and removal of samples from the moulds as to allow optimum reproducibility of results.

Curing time (or conditions) might have to be modified to allow complete carbonation of lime.

We think, however, that it is possible, on the basis of the data available, to start a discussion on suitable criteria for selection.

Some suggestions on such criteria are, therefore, offered here below, it being intended that they are to be considered as extremely tentative, a starting point for discussion and not the draft for specification.

Workability

Mortars should be tested in a condition of optimum workability. It is obviously desirable that such a condition be measurable in an objective and quantifiable way.

Setting time

Three days maximum (although for some applications a slow setting mortar, ten days maximum, could be tolerated).

Compressive strength

The mortars should not be much stronger than the ones used in the old masonry. As compressive strengths of 2.2 - 2.9 MPa were reported for old brick masonry in good condition (Laner, 1979) and such masonry would have a sizeable strength even if the mortar had no compressive strength at all (Guidi, 1966) we have come to the conclusion that a 0.5 to 3.0 MPa range is advisable.

Flexural strength

It is desirable that this be reasonably large although not exceedingly so. An interval 0.4 to 2.5 MPa might be suitable.

Modulus

This could hardly be a selective test in the present state of things. The execution is rather costly and results are not very indicative.

Porosity

20% minimum, with at least 65% above 0.1 μ. This, however, is a very moot point and could be decided only after a lot of measurements of water vapour permeability.

Water absorption

A minimum value of water absorption, compatible with the selected porosity values, should be specified. This would ensure exclusion of hydrophobic mortars when the latter quality is to be considered objectionable.

This provision, however, could be waived and, on the contrary, a maximum value of water absorption could be specified when hydrophobicity is considered desirable (e.g. for a rendering on a damp wall) provided that it is accompanied by the specification of a minimum value of water vapour permeability.

We are not in a position to suggest figures for such minimum and maximum values at the present moment.

Water vapour permeability

A minimum value should be specified in all cases. We cannot suggest any figure now because no measurement was taken by us.

Alkaline elements

Extractable alkaline elements should be as low as possible. An upper limit of 8 meg/kg (that is about twice the amount extractable from lime mortars) would allow the use of five of the mortar formulas tested by us (besides lime/sand). Bringing down the limit to 7 meg/kg would already leave only two formulas in the game.

Increasing the upper limit to 12 (three times lime mortar) would allow the use of lime/Portland Cement (low in cement) and lime/pozzolana (low in pozzolana).

We think that a maximum of 8 meg/kg might be a reasonable compromise.

According to a set of criteria we have outlined, the following mortars, among those tested in the present project, would be approved :

Slow setting

Lime putty/sand (L_1 S_3), with or without admixtures, slow setting

Lime/brick dust/sand (L_1 B_1 S_3) with water-reducing admixture, slow setting.

Rapid setting

Lime/cement, low alkali/sand (L_3 Cla_1 S_{12}) without water-reducing admixture.

Obviously additions to this list could easily be made by modifying mortars which do not qualify according to these criteria.

For instance, hydraulic lime mortars(Lh_1 $S_{1.6}$) which do not qualify because of excessive strength (but show a low content of alkaline extractable element) could be easily brought within the limits by the addition of lime.

ANNEX 1

A. Materials

Binders

Hydrated lime powder :	Calce ventilata in polvere Italcementi, Marcellina, Italy
Hydrated lime putty :	Grassello di calce Armando Piccini, Via Portuense, Roma, Italy A thermogravimetric analysis indicated approximately 50% (w/w) of water, Ca $(OM)_2$ approximately 45% (w/w) and $CaCO_3$ approximately 5% (w/w)
Portland Cement :	Cemento 325 Italcementi, Colleferro, Italy
(Portland) Cement Artificial (low alkali content)	CPA 55 HTS (haut teneur en silice) Ciments Lafarge, St. Cloud, France
Hydraulic natural lime :	Chaux blanche (L^{++}) Ciments Lafarge, St. Cloud, France
Hydraulic lime :	Calce Idraulica Italcementi, Bergamo, Italy

Hydraulic action aggregates

Pozzolana :	Pozzolana superventilata Pozzolane di Salone, M. Testa, Via di Salone, Roma, Italy
Calcium carbonate :	Carbonato di calcio, polvere leggera $CaCO_3$, 98.5%. Soluble alkali content 1.12% Codex, Carlo Erba, Milano, Italy

Brick dust :	Polvere di mattoni Mercuri, Piazza dei Satiri 47, Roma, Italy
Sand :	Sand for the preparation of plastic mortar specimens, with grain size distribution according to the Italian standards(Decreto Ministeriale 3 giugno, 1968) It is a siliceous sand, with round grains, of natural origin, from the shore of Lake Massaciuccoli. It is sold in bags of 1.350 Kg. weight (the amount suitable for the preparation of three mortar samples) by S.I.S.A., Torre del Lago, Italy An X-ray diffraction analysis (S.Z. Lewin, unpublished data, private communication) shows that the main components of the sand are : α-quartz and a plagioclase (probably sanidine)
Marble powder :	Polvere di marmo Scazzocchio e Belardi, Via Capo d'Africa 1, Roma, Italy

<u>Admixtures</u>

Sodium gluconate :	Gluconato di sodio, 98% Carlo Erba, Milano, Italy
Rheobuild 561:	Cement admixture, water reducing agent, according to ASTM 494 type A, Mac Modern Advanced Concrete, Via Taro 3, Roma, Italy

Fluid st :	Standard type, water reducing agent for concrete Ediltecnica, Via dei Monti Tiburtini 518, Roma, Italy

Water

Tap water :	Rome water has a hardness of 30 french degrees

We wish to express our gratitude to the firms : Ciments Lafarge, Italcementi, Mac Modern Advanced Concrete and Ediltecnica, which donated cements and admixtures used in our experiments.

B. Equipment

Balance	Technical balance by Sartorius, Type 1104, 0.1 g. precision, maximum load 1000 g.
Automatic mortar mixer	Tray capacity 4.7 l. RMU, Giazzi, Via Grumello 57, Bergamo, Italy
Enbloc, settling machine	RMU - Giazzi
Moulds	Stainless steel, for the preparation of three specimens (4 x 4 x 16 cm.) RMU - Giazzi
	PVC, for the preparation of 6 samples (5 x 5 x 5 cm.) Istituto Centrale del Restauro
Humidity cabinet	Stainless steel and glass, home made, R.H. 90 $\pm$ 5%
Hair thermohygrograph	Type 252 UA, Lambrecht
Vicat needle	Type L26, Controls, Milan, Italy
Thermohumidifier	Defensor 4000V

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X-RAY DIFFRACTION AND SCANNING ELECTRON MICROSCOPE ANALYSIS OF CONVENTIONAL MORTARS

S.Z. LEWIN [+]

SUMMARY

Lime-sand, lime-pozzolana-sand, lime-cement-sand mortars were studied through the examination of the separate components and mortar samples prepared under controlled conditions, after a few months' ageing.

Extensive lime carbonation and weak lime-sand adhesion can be explained by SEM pictures.

Transformation of hydrated calcium silicate gels into tobermorite crystals is an important factor in the ageing of hydraulic mortar mixtures and may offer some hope for an approximate determination of age.

Even if XRD and SEM alone are unable to provide an adequate characterization of the mortars, their use in the study of setting and ageing reactions can produce results of high interest.

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INTRODUCTION

Although the elemental analysis of cements, concretes, plasters and mortars constitutes a well-established methodology (1) there are many instances when a more species-specific type of information is needed. That is, the compositions of these masonry materials are commonly expressed in terms of their hypothetical equivalent content of CaO (generally represented in the literature of this field by the symbol c), SiO_2 (s), Al_2O_3 (A), Fe_2O_3 (F), H_2O (H), CO_2 (C), and SO_3 ($\bar{S}$). But such analytical data does not reveal, e.g., how much of the Ca-content is present in the form of free lime (C), or the hydroxide (CH), or calcite ($C\bar{C}$), or gypsum ($C\bar{S}H_2$), or the various silicates, aluminosilicates, ferrites, etc. Also of interest is the degree of crystallinity or lack thereof of these various phases.

Information about the presence and relative proportions and textures of these and other phases in various masonry materials as a function of the types of ingredients employed, the manner of preparing the end product, and the conditions that prevailed during the initial cure and the subsequent exposure to the environment would be of value in a number of applications, and in particular in the field of the preservation and conservation of historic or artistic works.

For example, it would be of significance in the investigation of old or ancient structures with respect to characterizing the raw materials and technology that had been employed by their makers. It would improve the reliability with which restored parts of structures can be distinguished from the original remainders. It would contribute to objectivity in estimating the age and need for conservation of parts of edifices and monuments. It would guide the restorer in selecting repair and replacement materials that are historically correct and chemically compatible with the surviving masonry. And, perhaps most importantly, it would help elucidate the mechanisms of deterioration of exposed masonry, and provide the tools for assessing the effectiveness of measures taken for the purpose of slowing or stopping the decay processes.

The insights that stem from the combination of (a) X-ray diffractometry (XRD) and (b) scanning electron microscopy (SEM) with (c) elemental analytical data can provide some of the species-specific information alluded to in the foregoing. In this paper, these techniques are applied to the ingredients and mixtures typical of mortars used in historic edifices during the past several centuries. Powder XRD reveals which crystalline phases are present in substantial concentrations (generally ca. 5% or greater). SEM shows their characteristic crystal growth patterns, and their textural properties, by which is meant the size distribution, degree of perfection and/or orientation, and the pattern of intergrowths within individual phases and between different phases that is responsible for strength development during curing and ageing.

EXPERIMENTAL

The raw materials utilized for producing the classical-type mortar mixtures consisted of: (a) lime putty, (b) portland cement, type I, (c) low alkali portland cement, (d) sand, (e) pozzolana, and (f) hydraulic lime. These were typical commercially available materials, and were obtained from local builders' supply firms in Italy. These ingredients, as well as the standard mortar compositions made with them, were provided for this work through the kindness of Dr. G. Torraca and Miss S. Peroni of the International Centre for Conservation at Rome (ICCROM).

The lime putty, portland cements, and hydraulic lime were studied in the air-dried state, as well as in the fully hydrated and cured condition. The same was done for binary mixtures of each of these components with (1) sand, and (2) pozzolana, and then with ternary mixtures of two types, viz. (3) lime + pozzolana + sand, and (4) lime + cement + sand. Table I shows the systems studied, and the types of data obtained.

A. Lime Putty

The lime putty employed gave the x-ray pattern of crystalline portlandite, $Ca(OH)_2$, with no indication in the low-angle scattering region of any significant amount of non-crystalline material (i.e., with particle sizes less than ca. 100 A). Minor diffraction peaks occurred at 3.05 and 2.92 A; these could be due to a small proportion of one or more of the C-S-H gels (2), and the former peak might arise in part from about 1 - 2% of calcite. The absence of any other peaks of these species precludes any more definite identification.

The scanning electron micrographs of the lime putty (Fig. 1 and 2) exhibit the thin, hexagonal plates characteristic of $Ca(OH)_2$; these have a maximum dimension of about 2 μm, and range downward in size to about 0.07 μm for the majority of the crystallites.

It is significant that the portlandite content of this lime putty is so clearly recognizable in both XRD and SEM, for as will be seen in the following, it is no longer detectable by these techniques as a major constituent of a number of the binary and ternary mortar formulations that contained this lime putty as a principal component.

TABLE I. DATA OBTAINED FOR SYSTEMS STUDIED

System [+]	Proportions	XRD	SEM
L	---	Portlandite; minor calcium silicate hydrate	Fig. 1, 2
L + S	1:3	Calcite, Quartz, Feldspar	Fig. 3, 4
L + P	1:1	Portlandite; Calcium Aluminate Hydrate; Calcium Silicate Hydrate; Pozzolana (upon ageing: hydrocalcite; prehnite)	Fig. 5 - 8
L + P + S	4:1:10	Calcite, Quartz, Feldspar	Fig. 9, 10
C, or l.a.C	---	Alite; Belite; C_3A; Ferrite; Calcite; trace of Anhydrite	Fig. 11, 12
C + H	1:2	Calcite; Tobermorite; (upon ageing: Afwillite)	Fig. 13 - 16
L + C + S	3:1:10	Calcite; Quartz; Feldspar	Fig. 17 - 20
L + C + S	4:1:10	Calcite; Quartz; Feldspar	
L + C + S	3:1:12	Calcite; Quartz; Feldspar	
L + C + S	4:1:12	Calcite; Quartz, Feldspar	
C + P	1:1	Tobermorite; Calcite; Calcium Aluminate Hydrate; Quartz; Feldspar	
hL	---	Alite; Belite; C_3A; Ferrite; Calcite; Portlandite	
hL + S	1:3	Portlandite; Calcite; Quartz; Feldspar	Fig. 21 - 23

[+] L = Lime Putty; hL = Hydraulic Lime; C = Portland Cement; l.a.C = low alkali Portland Cement; P = Pozzolana; S = Sand.

B. Lime Putty + Sand

The sand used for these archetypical mortars consisted of small (ca. 1 - 2 mm) particles which XRD showed to be 90 - 95% a-Quartz, with the remainder a potassium feldspar that most closely resembles high sanidine (sodium potassium aluminum silicate, ASTM # 10 - 357).

Lime putty was mixed with this sand and water to a consistency that could be shaped into cubes, and these were allowed to cure at 90% R.H. for 7 days, followed by 53 days at 75% R.H. The cubes were then stored at 50% R.H. for one year before the present analyses were carried out on them. The curing, ageing, and storage were at ambient temperature (ca. 20°C).

XRD revealed no trace of portlandite in this mixture. The only crystalline phases found were calcite, quartz, and the feldspar component of the sand grains. SEM showed the hexagonal-plate-like morphology of the portlandite to have completely disappeared; in its place were masses of irregular particles partially fused into larger agglomerates (Fig. 3), which XRD showed to be calcite. The surfaces of the sand grains contained areas where these agglomerates adhered, and other areas where they did not; instead, at the latter sites numerous individual, tiny (0.2 - 0.6 μm) calcite crystallites had grown onto the siliceous substrate (Fig. 4).

It appears that there is very little interaction between calcium hydroxide and a quartz/feldspar substrate under the conditions of these studies. Instead, the principal chemical reaction occurs during ageing, and consists in the conversion of portlandite into calcite through the action of atmospheric carbon dioxide and water vapour.

Roberts (3) has pointed out that in the case of concretes the rate of carbonation at ordinary temperatures is greatest at a relative humidity in the region of 50 to 75%. At lower humidities very little carbonation takes place due to the lack of free water. The presence of large amounts of moisture also hinders the reaction by filling pores and reducing or preventing access of the CO_2 to the interior.

The mechanical weakness of this mortar mixture is due to the limited amount of interparticle fusion that develops at the calcite-to-calcite contacts (Fig. 3), and to the small affinity of the calcite crystallites for the quartz surfaces (Fig. 4).

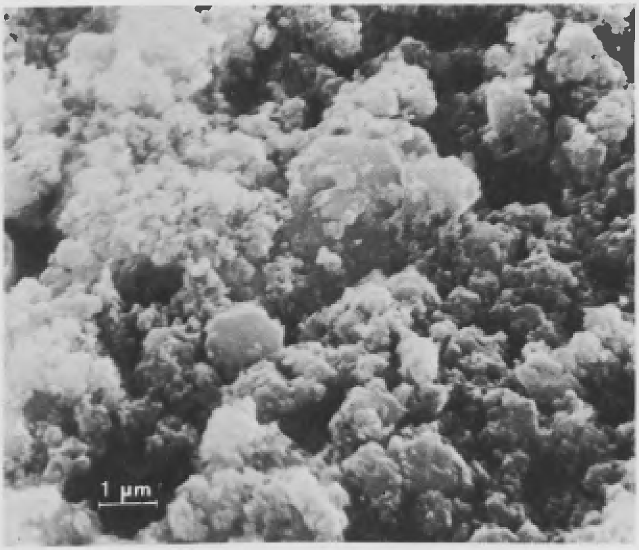

Fig. 1 Lime putty, showing relative proportions of well-developed hexagonal plates and small, irregularly shaped masses of $Ca(OH)_2$ crystallites.

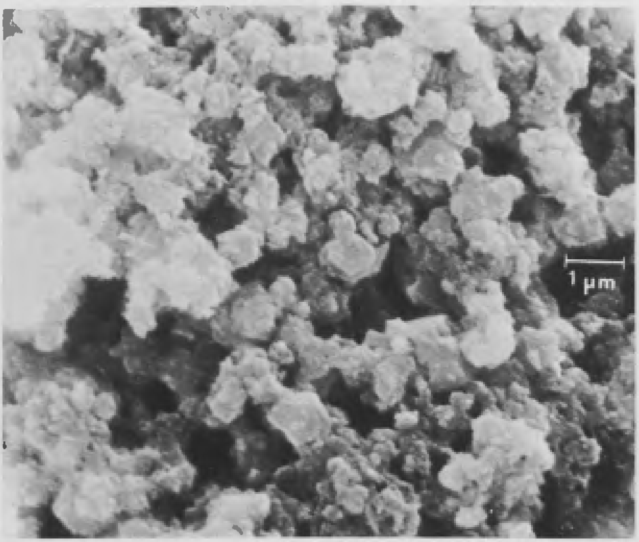

Fig. 2 Lime putty, showing the average particle sizes of the relatively well-developed portlandite crystals.

C. Lime Putty + Pozzolana

Pozzolana is a finely powdered silicate selected for its ability to interact with hydraulic constituents of mortars or cements (4), and as such might be expected to be reactive toward calcium hydroxide. The pozzolana employed in the present study came from the quarries of Salone, Italy. A typical chemical analysis was : 46.7% SiO_2, 11.1% Fe_2O_3, 16.7% Al_2O_3, 9.8% CaO, 3.7% MgO, 6.5% K_2O, 1.9% Na_2O, 2.4% volatile on ignition. About 90% of the particles were in the range of 1 - 5 µm in dimensions. XRD showed it to be largely glass, but with ca. 10 - 20% of crystalline components. Reference data, as well as comparison with authentic mineral standards showed these to be leucite, $KAlSi_2O_6$ (ASTM #15 - 47) and prehnite, $Ca_2Al_2Si_8O_{10}$ (ASTM # 7 -333). In addition, there is a small amount, ca. 3 - 5% of yoderite, a magnesium aluminum silicate (ASTM #12 - 625).

Lime putty mixed 1:1 with pozzolana, and with water sufficient to make a paste, was allowed to air-dry, and was then cured at 90% R.H. for 7 days. XRD showed the portlandite of the lime putty to be essentially all still present. However, minor amounts of calcium aluminum hydrate, $Ca_4Al_2O_7.xH_2O$ (ASTM # 2 - 007) and calcium silicate hydrate $5Ca_2SiO_4.6H_2O$ (ASTM # 3 - 0248), reaction products resulting from the interaction between the portlandite and pozzolana, were found to be present. SEM shows that the portlandite tends to adhere to and thoroughly coat the glassy pozzolana shards (Fig. 5); the calcium aluminate and silicate reaction products occur as chunky, polygonal crystallites growing out of the pozzolana surface (Fig. 6).

Upon further curing (2 months), very pronounced structural changes occur. SEM shows that the pozzolana surfaces have been extensively etched, producing a greatly increased internal surface area (Fig. 7 and 8). The portlandite is no longer evident in its original morphology in the micrographs, yet its diffraction pattern is still strong. Hence, it appears to have undergone recrystallization and to have become incorporated into the network of dense masses bridging the pozzolana shards (Fig. 7). This provides a rationale for the well-known effect of pozzolana in improving the quality of mortars (5).

XRD also shows the 2-months-aged specimen to contain an increased amount of prehnite, as well as a small amount (ca. 5%) of monohydrocalcite, $CaCO_3.H_2O$ (ASTM # 22 - 147).

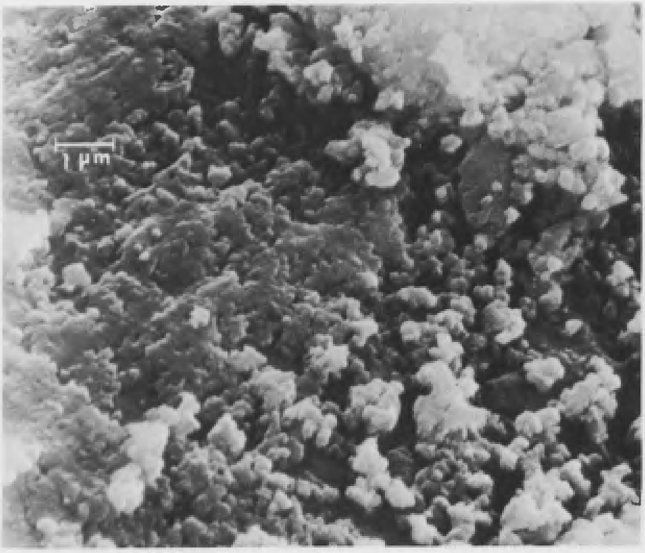

Fig. 3 Lime + sand mortar. Surface of a sand grain covered with an agglomeration of irregularly shaped calcite crystals.

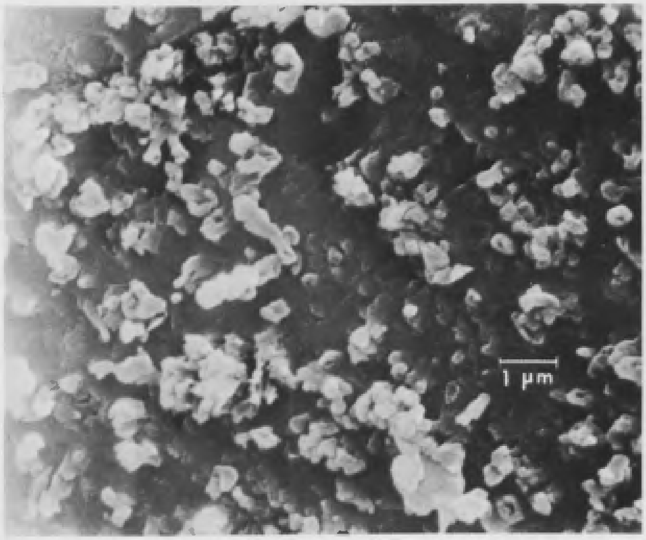

Fig. 4 Lime + sand mortar. Surface of a sand grain with individual calcite crystallites attached.

It is significant that in the lime putty-sand mixtures substantially all the portlandite had converted to calcite during the initial ageing period, i.e., the first two months, whereas in the lime putty-pozzolana mixtures, it had not. Two conclusions follow from these observations: (1) The portlandite in lime putty is inherently readily reactive toward atmospheric CO_2, and (2) The rate of reaction is primarily limited by the porosity, i.e., gas permeability, of the mortar. The very fine particle size of the pozzolana results in a much less permeable mortar than that in which sand is the filler.

D. Lime Putty + Pozzolana + Sand

A mortar was prepared in which the proportions by weight of lime putty, pozzolana, and sand were 4:1:10. These ingredients were mixed with only enough water to yield a workable mass that could be shaped into cubes. The cubes were cured and aged as described in B above.

XRD showed only calcite, quartz, and feldspar. SEM disclosed that the calcite was present as agglomerates of tiny particles, 0.06 to 0.3 µm, coating the pozzolana shards, as well as some areas of the sand grain surfaces (Fig. 9). Other areas of the quartz grains were relatively bare, with individual tiny calcite crystals scattered thereon (Fig. 10). Because of the small reactivity of portlandite or calcite with quartz surfaces, the calcite-coated pozzolana shards were weakly bonded to the sand grains. The relatively high proportion of sand in this mortar produced a permeable matrix that was conducive to the atmospheric conversion of the hydroxide to the carbonate.

Thus, it appears that the addition of a small proportion of pozzolana to a lime-sand mortar does not have a significant effect in improving its strength or ageing characteristics.

E. Portland Cement

Two commercial grades of portland cement were used in these studies: type I, and low alkali. Both were found by XRD to contain as their major components C_3S (ca. 50%), β - C_2S (25%), C_3A (10%), and the calcium ferrite solid solution phase (composition between C_2F and C_6A_2F) (10%), plus minor proportions of $C\overline{C}$, $C\overline{S}$, MgO, Na_2O, and K_2O. The characteristic morphology of the cement

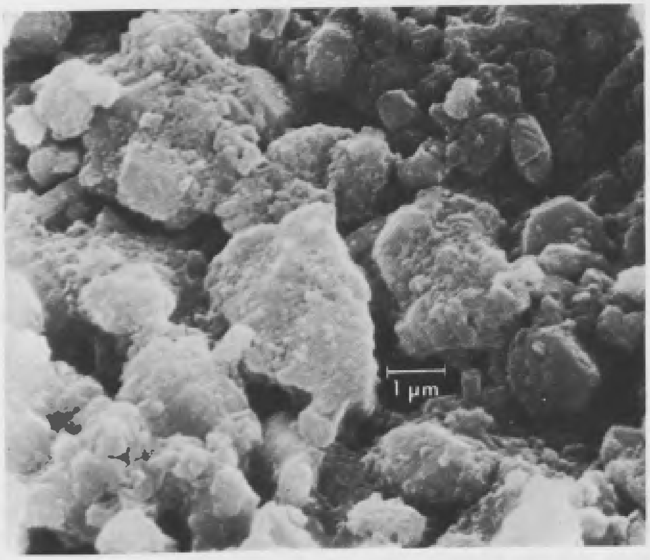

Fig. 5 Lime + pozzolana, 1:1, aged 7 days, showing the individual glass shards with much smaller portlandite crystals covering their surfaces.

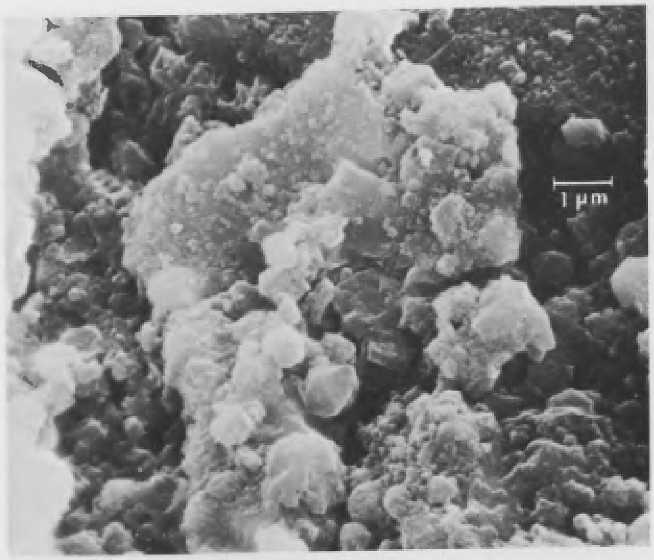

Fig. 6 Lime + pozzolana, 1:1, aged 7 days, showing glass shards with chunky, polygonal crystals of aluminate and silicate reaction products on surface.

Fig. 7 Lime + pozzolana, aged 2 months. The glassy shards are etched and covered with platy growths that bridge across from shard to shard.

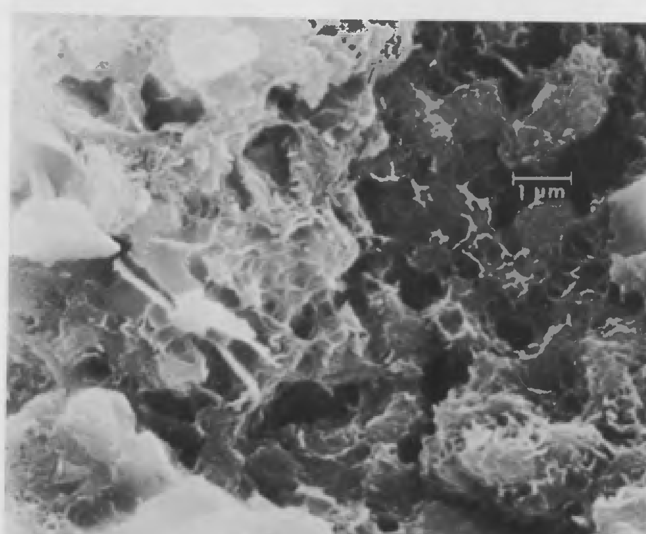

Fig. 8 Lime + pozzolana, aged 2 months. The platy growths on the glass shards contain the original portlandite.

particles is shown in the micrographs of Fig. 11 and 12. The cements differed primarily only in their elemental content of alkali and aluminum.

When made into a thin paste with water, and then allowed to air-dry during 2 days, followed by curing at 100% R.H. and 25°C for six weeks, the principal reaction products were found by XRD to be calcite, tobermorite, and afwillite. There was very little, if any, portlandite (as is shown below, a small proportion of portlandite is not unambiguously identifiable in the presence of the other constituents).

The C-S-H phase or phases that are among the principal initial products of the reaction of portland cement ground clinker with water (6, 7), and that frequently occur as a colloidal gel (8 - 12), were found to have developed in the present case into a highly crystalline product that gives a sharp, intense x-ray diffraction pattern. The observed peaks agree well with those calculated on the basis of an orthorhombic unit cell that has parameters close to those reported for the mineral forms of tobermorite, $5CaO.6SiO_2.2\frac{1}{2}H_2O$ (ASTM # 10 - 374). The crystal structure of tobermorite appears to be somewhat variable (13 - 15); the ASTM data file lists at least three diffraction patterns, corresponding to three somewhat different orthorhombic unit cells. The present x-ray data, together with those of the mineral forms, are given in Table II. The strongest peak is at d = 4.95, corresponding to reflections from the (004) planes; the next most intense peaks occur at 3.06, 7.6, and 2.64 A.

TABLE II. XRD IDENTIFICATION OF TOBERMORITE IN PORTLAND CEMENT-WATER REACTION PRODUCT

hkl	Calculated[+]	Observed (this work)
002	9.8	9.9
010	7.4	7.6
102	7.35	7.30
111	5.87	5.90
012	5.91	
200	5.6	5.7
004	4.90	4.95
202	4.83	4.77
210	4.44	4.44
020	3.70	3.82
022	3.46	3.48
220	3.08	3.06
221	3.04	
224	2.61	2.64

[+] Based on a unit cell having $a_0 = 11.1$, $b_0 = 7.4$, $c_0 = 19.6$.

ASTM gives the following parameters for three naturally occurring minerals :

# 10 - 374 :	$a_0 = 11.12$,	$b_0 = 7.32$,	$c_0 = 19.2$
# 19 - 1364	11.27	7.35	22.74
# 6 - 5	11.24	7.30	28.0

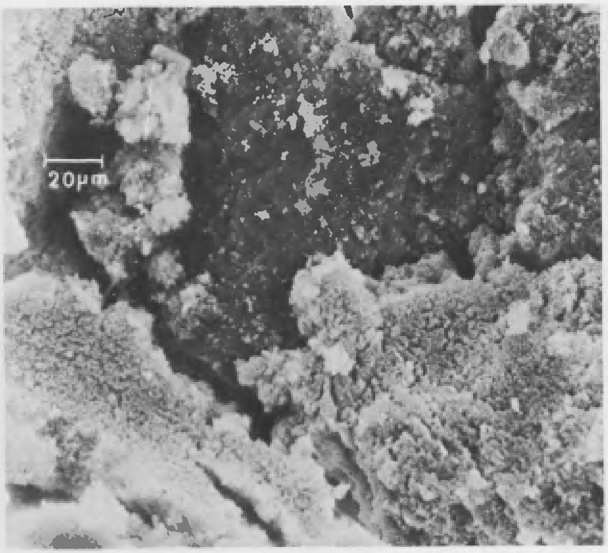

Fig. 9 Lime + pozzolana + sand. The dark area is a sand grain, part of which has massed agglomerates of calcite particles resting on it. The other, light areas are pozzolana shards covered with calcite crystallites.

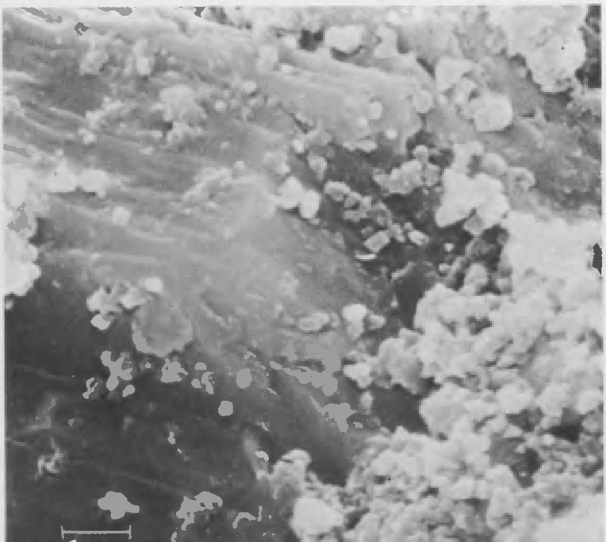

Fig. 10 Lime + pozzolana + sand. Detail of the relatively bare surface of a sand grain. The small crystals on its surface are calcite.

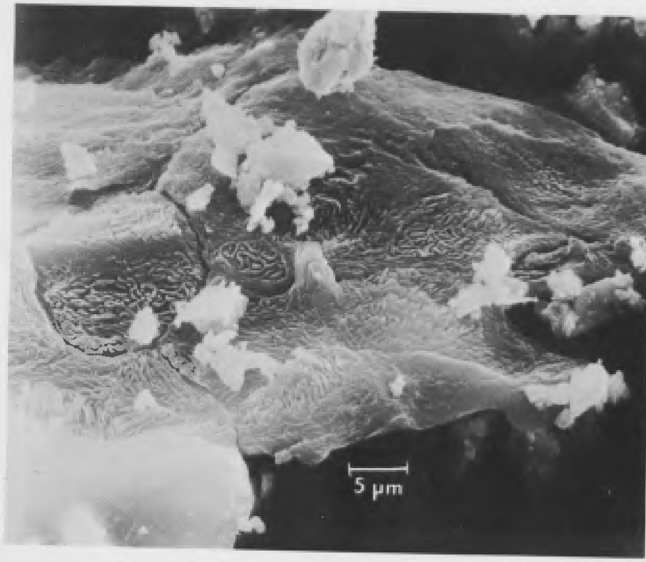

Fig. 11 A particle of Portland cement ground clinker.

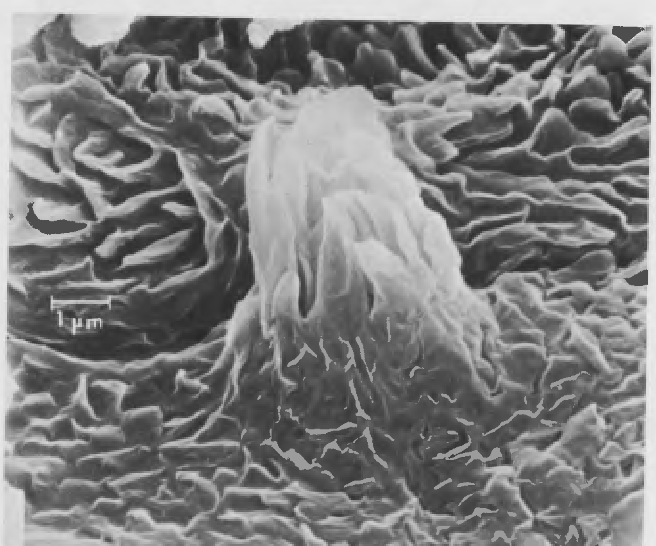

Fig. 12 Detail of a clinker particle, illustrating the sintered, porous morphology resulting from the high temperature kiln reactions.

The colloidal or gel-like forms of C-S-H give electron diffraction maxima corresponding with the (220), (400), (040), (620), (440), (800), and (260) spacing of crystalline tobermorites, but all (002) spacings are absent (2). In the present case, (002) and (004) are present as strong peaks, confirming the non-gel character of the tobermorite.

The two strongest peaks of portlandite are at 4.90 and 2.65 A, and hence are overlapped by those of tobermorite. The strongest peak of calcite is at 3.04, and is also overlapped by tobermorite. However, the presence of calcite is easily verified, even on a micro-scale, by chemical tests, viz., the evolution of CO_2 when treated with acid. There is no equivalent sensitive wet chemistry that can be applied to the detection of portlandite in the presence of the alkaline constituents present in mortars and cements. It is possible to extract $Ca(OH)_2$ from a mortar or cement by means of organic solvents, such as ethylene glycol (10) or a mixture of acetoacetic ester and iso-butanol (16), but these extractants tend to decompose C-S-H, and it is not possible to distinguish unambiguously between $Ca(OH)_2$ present as such, and that generated by the extraction process, particularly if the proportion is small.

Upon treating the specimen under discussion with 1 M HCl, added dropwise until effervescence ceased, the intensity of the 3.06 peak was observed to decrease to about 2/3 of its former magnitude. Hence, that peak does indeed belong in common to calcite plus tobermorite in this preparation.

The tobermorite results from the crystallization of the C-S-H gel. The latter is not generally identifiable by XRD in mortars and cements or concretes, for it only yields diffraction maxima in the neighbourhood of 3.05, 2.8, and 1.8 A, and the latter two are weak and broad (13). The 3.05 peak is strong and broad, and coincides with the strongest peak of calcite. Portlandite and quartz also have diffraction maxima that interfere with one or more of those of C-S-H. However, as the crystalline tobermorite forms in significant concentration, its diffraction pattern becomes clearly recognizable. In the present case, the principal peaks of tobermorite were observed to increase in sharpness and intensity as the duration of ageing at 100% R.H. increased.

In our portland cement-water mixtures, the x-ray diffraction pattern of tobermorite was very weak during the initial curing period (2 - 7 days), and grew progressively in sharpness and intensity during the ensuing six weeks of ageing at 100% R.H. Similarly, the

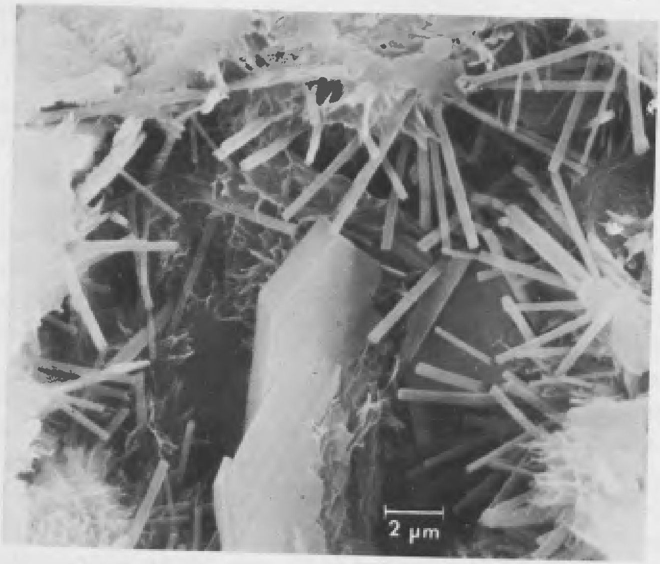

Fig. 13 Portland cement + water, cured 6 weeks. The fibres (tobermorite) grow out from the clinker grains. The hexagonal plates are either calcium aluminate hydrate or portlandite transformed into calcite.

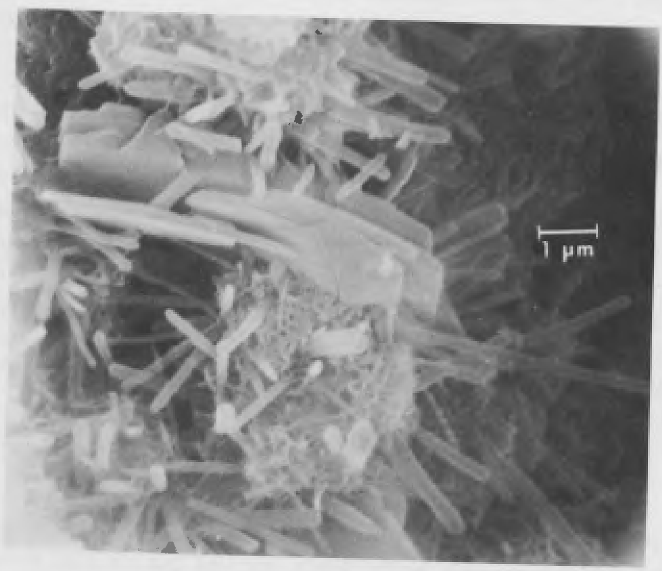

Fig. 14 Portland cement + water, cured, 6 weeks. The tobermorite fibres are solid, and several show hexagonal cross-sections.

x-ray pattern of afwillite became clearly evident only after the ageing period. Its intensity indicates that it is present as a minor constituent (< ca. 10%). The mineral, afwillite, ASTM # 9 - 454, is $Ca_3 (SiO_3OH)_2 \cdot 2H_2O$, and has monoclinic symmetry. It is reported as occurring in cement pastes in the form of needle-like crystals (9, 14).

The SEM micrographs of this cement-water reaction mixture show four distinct types of crystal morphologies. (1) Needle, fibre, or rod-like crystals grow out from the original clinker grains. These tend to be long, straight, solid, and appear to have a hexagonal cross-section (Fig. 13, 14). (2) The surfaces of the clinker grains have acquired a sponge-like texture due to a profusion of thin platelets that have grown out perpendicular to the original surface, and in the process have etched away some of the substance of the grain (Fig. 15). (3) Relatively large, well-formed, hexagonal plates have grown in the spaces between clinker grains (Figs. 13, 14, 15). (4) Crystals showing the rhombohedral habit of calcite occur associated with some of the clinker grains (Fig. 16).

On the basis of the intensity of the x-ray pattern of tobermorite in these specimens, and the ubiquity of the needles and surface platelets, these features are identified as the growth pattern adopted by the tobermorite.

Walsh, et al (17) have reported a SEM study of type I portland cement/water preparations that were made and cured in a fashion very similar to the present work, and their SEM technique was likewise equivalent to ours. Their micrographs show essentially the same morphological features as ours. Purely on the basis of morphology, together with the assumed hydration chemistry of portland cement, they identify the fibres as C-S-H or "tobermorite gel" and the hexagonal plates as portlandite. No XRD data were reported. They also state that ".... the fibres have geometrical cross-sections which can sometimes be identified as square."

The present results differ in showing that at least a large proportion of the fibres are truly crystalline in character, and that there is little or no portlandite, despite the substantial proportion of the hexagonal platy crystals.

The calcium aluminate phases in hydrated cement typically form hexagonal plates which tend to form tabular arrays (15.18). This is very similar to the morphology observed in the present case. It has also been reported that the hexagonal plates of

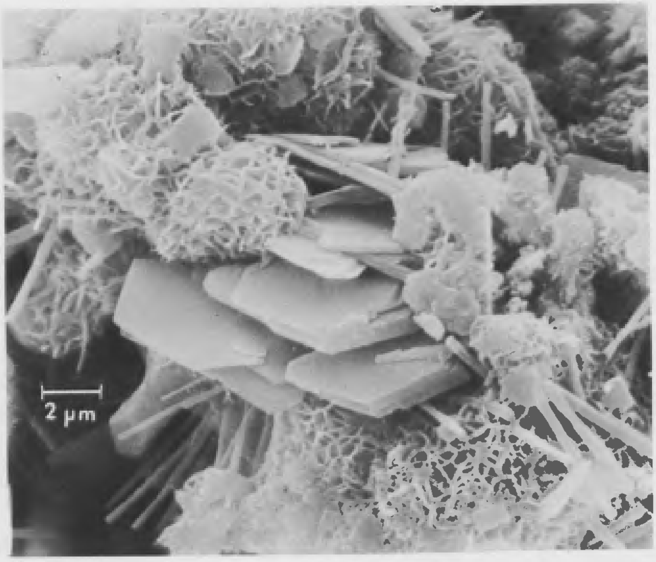

Fig. 15 Portland cement + water, cured 6 weeks. The clinker surfaces are covered with growths of platy crystals. The large, tabular plates have the morphology characteristic of the calcium aluminate hydrates.

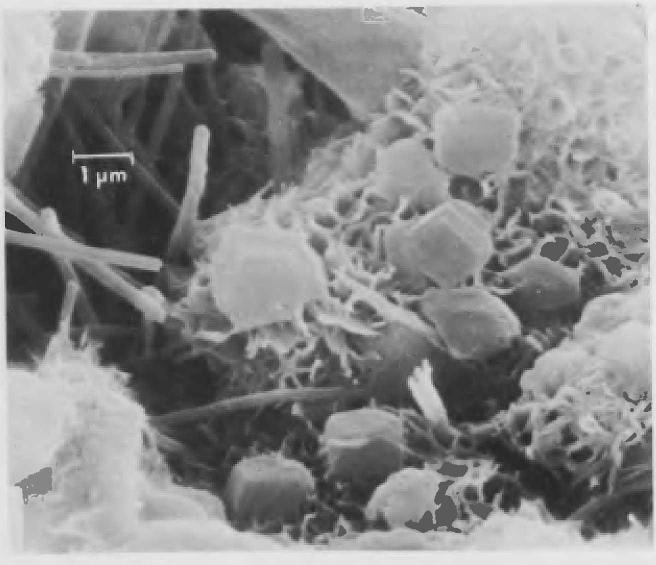

Fig. 16 Portland cement + water, cured 6 weeks. Occasional rhombs of well-developed calcite are encountered on the clinker grain surfaces.

portlandite can be transformed polymorphically into calcium carbonate without undergoing a change in their external shapes (10).

Several high magnification TEM investigations have been reported of C-S-H and tobermorite gel. In most of these, the particles were obtained by sawing the sample of set cement to produce dust, and then dispersing the particles ultrasonically in water prior to mounting on the TEM grid. The morphology thus seen has been reported as that of hollow tubes, or crinkled, crumpled foils (2, 6, 7, 18, 22, 23). However, there is some uncertainty as to whether the particles' morphology may have been altered by the rigorous conditions of TEM sample preparation. The crystalline form of tobermorite observed in the present work, derived from fracture surfaces subject to no further manipulation other than evacuation, is clearly a solid rod (Fig. 13, 14).

The rod-like crystals seem to exhibit a hexagonal cross-section, and the platelet growths on the clinker grains also appear to show a tendency toward a six-sided outline. This is not what would be expected for an orthorhombic crystal structure, such as tobermorite has, which under favourable growth conditions should give rise to crystals that exhibit rectangular shapes. It may, however, be that the apparently hexagonal crystal habits observed in these preparations were produced in the course of the sequence of solid state transformations that result in the eventual formation of tobermorite from the initially formed C-S-H gel-portlandite "eutectic". Grudemo (19) has demonstrated this type of occurrence in the topochemical transformation of $Ca(OH)_2$ into calcite, i.e., there is no change in crystal habit accompanying the carbonation.

The occurrence in our preparations of truly crystalline tobermorite rather than any of the C-S-H forms of hydrated calcium silicate may be related to the low proportion of $Ca(OH)_2$, for it has been shown (13, 15) that the tendency to form the crystalline varieties increases as the pH of the clinker grain surfaces decreases.

It appears, from these observations, that the presence or absence of crystalline tobermorite in a mortar is an indication of the chemical character of the cement used, and/or the method of preparation of the mortar (e.g. concentration and accessibility of atmospheric CO_2, or pH of the water, etc.), and not necessarily of the age or weathering history. It will be interesting to see whether an XRD-SEM study of ancient mortars discloses any trend in the type of C-S-H phase present.

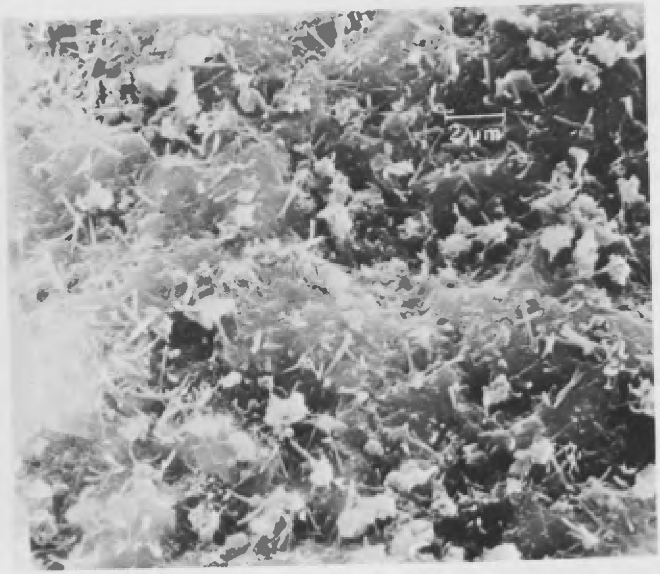

Fig. 17 Lime + portland cement + sand. The surface of a sand grain is here shown, containing a profusion of C-S-H or tobermorite fibres.

Fig. 18 Lime + portland cement + sand. The fibres can be seen to be directly bonded to the quartz surface.

F. Lime + Portland Cement + Sand

Various mortar specimens were prepared in which the proportions of lime putty to portland cement to sand were 3:1:10, 3:1:12, 4:1:10, and 4:1:12. After curing and ageing as previously described, these mortars were analyzed by XRD and SEM.

XRD showed the presence, in all cases, of only calcite plus the constituents of the sand grains, viz., quartz and feldspar. There was no evidence of portlandite, despite the substantial proportion of lime in the mixtures. This is consistent with the previous observation that when sand is the filler, the permeability of the mortar is sufficiently large to allow atmospheric CO_2 to convert the portlandite in the lime to calcite within several months of exposure (20, 21).

Since the cement component constitutes only 6% or less of these mortars, it is to be expected that its products will not be detectable in the x-ray diffraction pattern of the mortar. However, they are clearly recognizable in the SEM micrographs (Fig. 17 - 20). The surfaces of the sand grains contain a profusion of fibres or rods (Fig. 17), and most of these are bonded to the quartz, as can be seen in Fig. 18. This is the C-S-H and/or tobermorite phase, and its molecular attachment to the quartz (23) is the source of the mechanical strength contributed to these mortars by the cement component (7). It should be noted that the small size of these crystallites makes it difficult or impossible to detect them in mortars or cements by optical microscopy, as has been the experience of many who have sought them before the era of electron microscopy (8).

The lime component, which is no longer $Ca(OH)_2$, but has been converted into $CaCO_3$, shows nevertheless the characteristic crystal texture of the original $Ca(OH)_2$ in many places. This can be seen in the large, thin, hexagonal plates of Fig. 19. These are calcite, but in the form of pseudomorphs of portlandite. That is, in the course of the transformation of a crystal of $Ca(OH)_2$ into $CaCO_3$ under the conditions prevailing in these preparations, the internal crystal lattice rearranges, but the external shape is retained (19). Most of the portlandite of the lime putty was present originally as small, irregular crystallites, and these have been converted into the nondescript aggregates of calcite crystallites shown in Fig. 20.

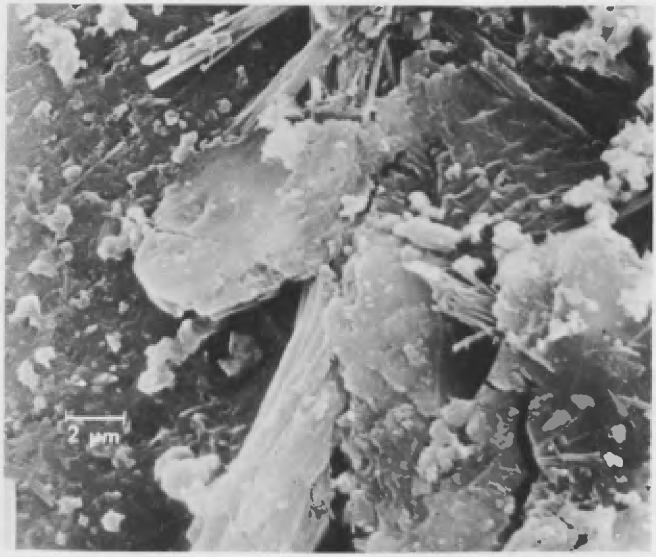

Fig. 19 Lime + portland cement + sand. The thin hexagonal plates are calcite, but retain the external shape of the precursor portlandite.

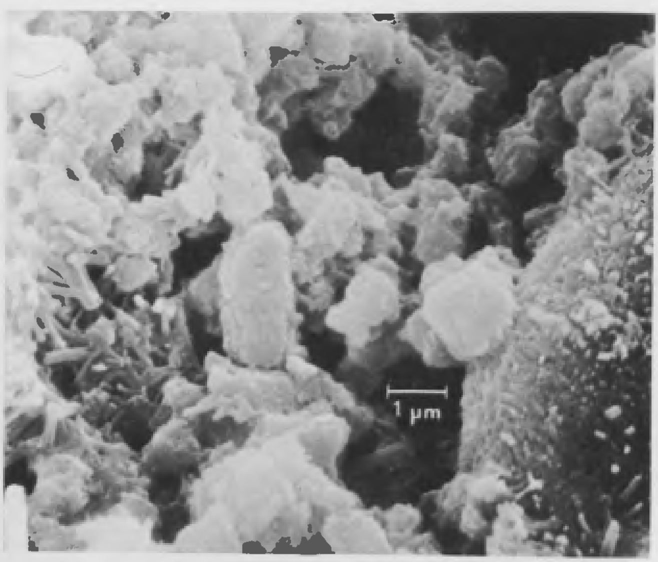

Fig. 20 Lime + portland cement + sand. The majority of the calcite is present as aggregates of tiny, irregularly shaped crystallites.

G. Hydraulic Lime + Sand

XRD shows the hydraulic lime used in these studies to consist of the mineral constituents of portland cement, plus portlandite, i.e., it is equivalent to a mixture of lime and cement clinker. When mixed with sand and water to produce a mortar, in the proportion of 1 part of hydraulic lime to 3 parts of sand, the result was similar to, but not identical with, that obtained from the lime + cement + sand mixtures. The principal difference is the presence of a substantial amount of portlandite in the cured, aged mortar from hydraulic lime whereas no portlandite remained in the similarly aged mortar made from lime + cement.

In the manufacture of hydraulic lime, the homogenization of the cement and lime phases is much more intimate than when these two ingredients are mixed by hand during mortar preparation; hence, in hydraulic limes the cement phase appears to coat and protect the portlandite from contact with atmospheric CO_2.

SEM shows that the surfaces of the quartz grains are covered with a felt-like mat of the hydration products of the cement (Fig. 21), out of which grow the fibres and rods typical of C-S-H and tobermorite (Fig. 22). The particles of cement clinker show the spongy, etched surfaces, with platelets and fibres growing out of them, typical of C-S-H formation (Fig. 22, 23). Large, thin, hexagonal plates that correspond to portlandite can also be seen (Fig. 21, 23).

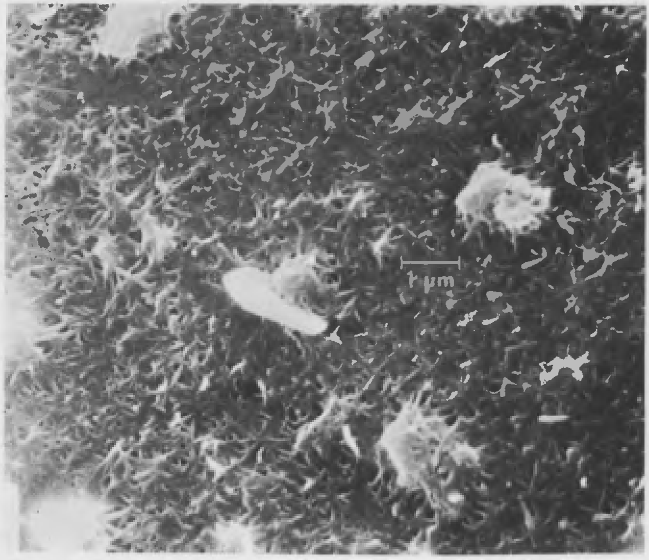

Fig. 21 Hydraulic lime + sand mortar. The surface of a quartz grain, covered a felted mat of C-S-H or tobermorite, with a crystal plate of portlandite in the centre.

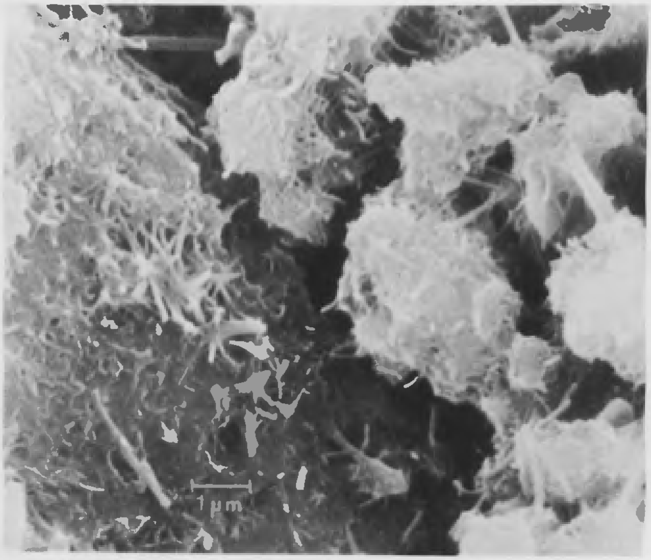

Fig. 22 Hydraulic lime + sand mortar. The surfaces of the clinker grains show the typical development of plates and fibres of the C-S-H phases.

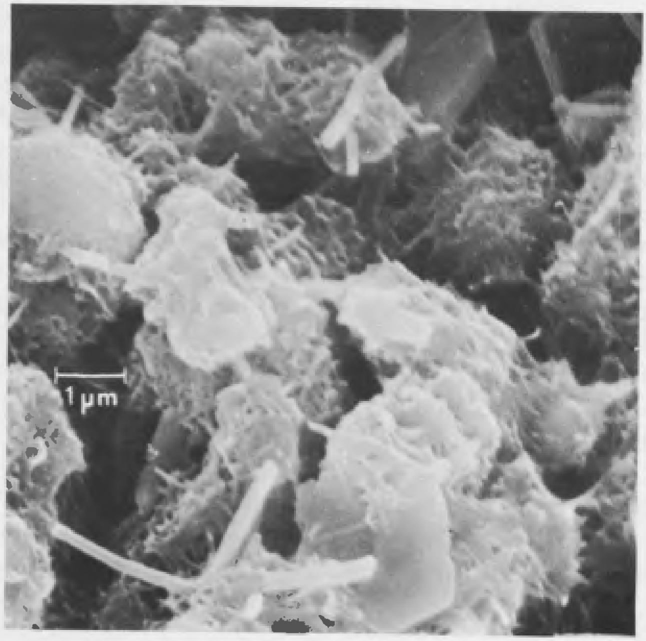

Fig. 23 Hydraulic lime + sand mortar. The C-S-H-covered clinker grains, tobermorite fibres, and thin, hexagonal plates of portlandite are characteristic of this mortar.

SUMMARY AND CONCLUSIONS

Analysis of the ingredients and mixtures employed in conventional mortars by XRD combined with SEM provides useful insights into the characteristics of these materials. It is shown that the portlandite of a lime mortar is converted into calcite within several months of exposure to atmospheric CO_2 at high relative humidities if the solid mass is permeable, as it is when sand is the filler. If the mortar is relatively dense, as when pozzolana is the filler, the rate of conversion to calcite is greatly reduced. If the calcium hydroxide crystallites are protected by intimate contact with cement, as in hydraulic lime mortars, portlandite persists despite exposure to CO_2 and H_2O vapour.

Because of these variable factors, determination of the carbonate content, or of the portlandite-to-calcite ratio, does not appear to offer any hope as a tool for estimating the age of the mortar in a historic edifice.

In the cured and aged mortars containing portland cement the occurrence of truly crystalline tobermorite is demonstrated. It will be of interest to look for the occurrence of this form of hydrated calcium silicate in samples of mortars of various ages, for the insights it may provide into the technology or exposure history of historic masonry.

Afwillite, portlandite, and calcite are also involved in the structure of the cured mortar, but their role appears to be minor.

It is evident that neither XRD nor SEM alone can provide an adequate characterization of these mortars. XRD gives species-specific information that is far more useful than the elemental ratios yielded by x-ray fluorescence (XRF) or classical wet chemical analysis. But XRD does not reveal much about amorphous or poorly crystalline constituents, such as C-S-H, and it is insensitive to small concentrations which may be of critical importance, such as the hydration products that form at the interfaces between cement and filler grains. SEM discloses the sizes, shapes, and textures of the internal structures in the mortar with remarkable definition and minimum distortion due to specimen preparation, but morphology is not an unambiguous key to composition. The same substance may develop with quite different crystal habits, e.g. fibres, laths, and platelets, and pseudomorphs may occur in which the external shape and symmetry of the crystal do not correspond to its internal lattice type. However, when used together, each of these techniques complements and supplements the other, and this type of integrated analysis can be expected significantly to advance our understanding of the characteristics of the masonry materials encountered in historic structures.

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ROME. ARCHITECTURE/COULEUR

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SUMMARY

This paper is meant as contribution to the understanding of colour significance in architectonical restoration and is based upon historical and technical evidence.

The identification of original chromatic relations in architectonical facings called for: 1) iconographical documentation carried out on paintings; 2) archives research work; 3) scientific and technical analysis.

Examples are given to show that while XV to XIX Cent. Roman buildings appearance was generally dependent on the colour of the specific building materials employed, analogous chromatic relations were sometimes simulated by facing mortars imitating such building materials as bricks and travertine.

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Les récentes polémiques sur quelques restaurations d'édifices monumentaux à Rome (+) ont fait ressortir la question de la définition des couleurs soit dans l'architecture soit dans le noyau historique de la ville entière. Tous, ou presque trous, les choix effectués montrent une grande variété, ou mieux un caractère evidemment arbitraire.

Dans la plupart des cas le choix est basé sur des évaluations subjectives, ou est dû à la possibilité plus facile et économique de trouver les matériaux de construction, ou bien à une évaluation qui ne tient pas assez compte de l'importance de la couleur dans l'aspect de l'architecture. Vérification faite, la signification visive d'un ancien monument, d'une route ou d'une place peut souvent en résulter dénaturée.

Dans la plupart des cas la couleur des enduits n'est pas un effet décoratif superficiel, mais plutôt le résultat d'un choix conscient, qui souligne par sa variété le différent relief qu'on donnait aux membrures architectoniques, en contribuant aussi à déterminer les diffétents plans de la spatialité d'une façade ou d'un monument dans son ensemble.

L'importance de la couleur pour ce qui concerne la définition de l'espace architectonique a été déjà considérée. Une autre fonction très important de l'enduit coloré est de protéger la structure même des murs de l'action aggressive des facteurs ambiants.

Les matériaux qui revêtent les édifices, les enduits en particulier, constituent une sorte de "surface à sacrifier" au contact direct avec l'extérieur. Afin de prouver cette assertion, il suffit d'observer ce qu'il arrive à un ancient monument tel que S. Stefano Rotondo quand, en obéissant à la vogue de la structure à vue, on a éliminé l'enduit extérieur. La paroi, privée de la protection de l'enduit, a été endommagée par la corrosion atmosphérique et l'humidité a pénétré jusqu'à l'interieur endommageant les fresques. D'autres fresques, moins endommagées, sont situées en correspondance d'un mur extérieur encore enduit.

Le fait que les enduits soient détériorables est lié à leur fonction protectrice. Cela détermine la nécessité de réfections periodiques, qui expliquent le changement de couleur au cours du temps. La "couleur de Rome" définie ou définissable abstraitment ou à priori, n'existe pas dans la réalité. La "couleur de Rome" est le résultat de différentes interventions qui se sont suivies au cours du temps. Est-il possible de déterminer les couleurs originelles d'un bâtiment, telles ques elles ont été conçues et réalisées par l'auteur du projet?

Schématiquement on peut indiquer trois types d'investigation possibles.

(+) Cette recherche a été déjà présentée sous forme d'un audio-visuel en mars - avril 1981 dans l'exposition "Roma. Istruzioni per l'uso" organisée par la Municipalité de Rome et l'association culturelle "Italia Nostra".

Le premier consiste à analyser les couleurs des édifices comme elles ont été représentées par les peintres des époques passées.

Le deuxième est l'examen des documents d' archives.

Le troisième consiste dans une série d'analyses de laboratoire.

Dans les vues où les peintres ont représenté édifices, places, routes ou parties plus étendues d'une ville, il faut juger d'une manière critique si l'artiste a reproduit fidèlement les couleurs des édifices ou s'il les a interpretes à son gré.

Un exemple de la dernière methode peut être donné par l'examen de la vue du Palazzo del Quirinale exécutée par Van Wittel vers la fin du XVII siècle. Dans le tableau, qui est conservé dans la galerie du Palazeo Barberini, les parties saillantes de l'architecture (cordons, tympans et bandeaux des fenêtres, bossage angulaire) sont de couleur travertin, tandis que la couleur des plans de fond est evidémment gris-azur. A présent la couleur du Quirinale n'a pas changé dans les parties en pierre, mais dans les plans de fond elle a été modifiée.

Le même discours vaut pour la vue de Villa Medici, de la même couleur gris-azur, dans le tableau de Van Wittel.

Une autre vue d'un monument romain est celle conservée à Palazzo Braschi, qui montre Piazza Montecitorio au XVIII siècle: on y voit le palais dans l'arrière-plan; à côté, à la façon de coulisse, les angles de deux édifices montrent des bossages angulaires en travertin et les plans de fond de couleur gris-azur. Dans l'ensemble les couleurs actuelles de la place sont bien différentes.

On va maintenant considérer la deuxième source d'information: les documents d'archives. Dans les projets d'exécution détaillés, cahiers de charge, comptes ou listes de payements, on indique souvent les matériaux, les techniques, soit pendant la phase de construction soit pour les successives interventions d'entretien qui confirment ou changent les couleurs originelles.

Après un soigneux dépouillement aux Archivi di Stato de Rome il a résulté qu'en 1665 un certain maître Bartolomeo Pedroni fut payé pour des travaux exécutés à Palazzo Spada. Entre autres, on indique que dans le préau du palais "on a enlevé la poussière le jour 18 d'août sur toutes les loges en bas, sur l'entrée et sur le préau au dessous de la corniche ... on a donné la couleur travertin à tous les bandeaux, arcades, piliers de la loge surnommée ... au dessous de la corniche".

Cela nous démontre qu'il était d'usage de couvrir les paraments pierreux d'une mince couche de chaux chargée d'un pigment qui simulait la pierre et dont la fonction était soit esthétique soit protective.

Le détail montre un pilier photographié à lumière légèrment rasante, qui conserve quelques traces de la couche de chaux de couleur travertin appliquée pour l'entretien.

Un autre témoignage précieux remontant à 1748 concerne une maison, proprieté de la confrérie de S. Caterina della Rota e dei Funari, situé à Via delle Coppelle. Il s'agit d'un projet de travail oü l'on peut lire: "Vous donnerez de fond en comble la couleur de l'air aux fonds de la façade extérieure de cette maison "a scomodo di scala"; au contraire, les bandeaux des fenêtres chantournées, les corniches courant tout autour seront teintes de couleur travertin". C'est donc une confirmation du même rapport chromatique que nous avons déjà remarqué dans la documentation iconographique de Van Wittel. Dè plus, comme la photo nous montre, cet édifice, qui heureusement a été epaigné, au moins jusqu'à présent, par les restaurations inconsidérées qui se font de nos jours, conserve encore le souvenir de la "couleur de l'air" qui est indiquée dans le document.

Au contraire, les couleurs actuelles de l'église de S. Maria dei Miracoli ont entièrement changé. En 1793 la façade qui donne sur la place devait résulter de "couleur palombino" (vraisemblablement une tonalité de gris).

La "couleur de l'air" caractérisait la partie du côté de Via del Corso; "sur la façade du côté de Ripetta" il y avait une "couleur de patine" plus difficilement définissable.

Un dernier et plus récent témoignage, de 1847, qui traite des travaux de restauration et d'agrandissement d'un édifice situé entre Via della Sapienza, Piazza Madama et Via della Corsia Agonale, indique lui aussi une gamme de couleurs différente de l'actuelle. Dans le document on parle d'une demi-teinte verdâtre et d'une "demi-teinte nanchin", peut-être le ton de la toile écrue. Des couleurs, pourtant, dont il n'y a plus de traces aujourd'hui.

Le troisième instrument d'investigation afin de déterminer le chromatisme originel des édifices et les changements qu'il a subis au cours du temps, prévoit l'intervention directe sur les matériaux constitutifs de l'enduit et des couches de peinture superposées. Nous allons l'expliquer par exemple de quelques essais effectués sur l'ensemble monumental de S. Michele a Ripa Grande. Il s'agit d'un édifice des premières décennies du XVIII siècle qui a subi des agrandissements postérieurs. Dans le cas du S. Michele toutes les couches de couleur sont superposées l'une sur l'autre, ce qui permet de déterminer leur succession précise. Une analyse simple, qui pourrait devenir une pratique ordinaire et préliminaire dans beaucoup d'interventions de restauration architectonique, consiste à prélever un petit échantillon d'enduit coloré (pris, dans le cas présent, de la cour delle Zitelle), à partir des couches les plus superficielles jusqu'à la structure de la maçonnerie; puis l'échantillon, inclus par un traitement particulier dans une résine polyester, est observé, au microscope minéralogique grossi de 70 à 100 fois, et successivement photographié. On obtient de cette façon l'indication de la succession chronologique: de la couche la plus ancienne jusqu'à la couche visible. Dan ce cas, toutefois, la succession des couches, par exemple dans les bandeaux des fenêtres, a trové une confirmation plus évidente après l'ouverture d'une petite échelle transversale au moyen d'un soigneux nettoyage mécanique avec un bistouri: on a ainsi fait apparaître toutes les différentes couches et les passages chromati ques: de la couleur travertin originelle jusqu'au dernier rouge intense. Sur les plans de fond la section révèle en parallèle des successions analogues et la petite échelle met en évidence tous les tons, de la "couleur de l'air" jusqu'à la dernière couche jaunâtre. Un essai de nettoyage plus étendu, effectué sur la corniche et sur le plan contigu de la paroi, souligne encore plus évidemment le rapport chromatique originel.

Il est possible, par des investigations plus poussées, par exemple l'analyse diffractométrique ou l'usage du microscope à balayage, d'indiquer la composition des éléments constitutifs des pigments et des mortiers afin de parvenir à identifier, dans quelques cas avec une bonne approximation, l'époque probable des différents revêtements. A ce propos, l'Istituto Centrale del Restauro a mis au point une méthodologie spécifique à utiliser dans les cas les plus importants et complexes.

De toute façon, les seules indications données par les sections stratigraphiques et par les échelles de nettoyage ont permis de déterminer la succession soit des couches (au moyen des sections), soit de leur ton (au moyen des échelles), de sorte qu'on a pu reconstituer "graphiquement" l'aspect originel du S. Michele.

Lorsqu'on utilise les trois méthodologies illustrées il est possible d'identifier sans aucun doute, non seulement la coloration originelle de chaque édifice, mais aussi la succession de toutes les couleurs qu'on y a appliquées au cours du temps. Cependant une semblable recherche pourrait résulter en quelques cas longue et coûteuse, puisqu'elle exige en outre l'emploi de spécialistes et surtout d'instruments qui ne sont pas facilement disponibles. Il est toutefois possible, dans un très grand nombre de cas de reteinture, de déterminer les colorations correctes sans recourir à des examens approfondis sur chacun des édifices, surtout si l'on considère l'extension du centre historique de Rome (1.500 hectares), qui est un des plus grands du mond. On peut en effet indiquer quelques éléments constants qui, compte tenu des variations possibles, se présentent fréquemment dans les matériaux de revêtement employés pour le finissage des édifices.

Une première donnée très fréquente dans la coloration des édifices, au moins au cours de la période historique ici considérée (de la fin du XV siècle jusqu'à l'unification de l'Italie), est une partition des surfaces architectoniques basée sur deux tons chromatiques fondamentaux: l'un qui identifie les plans de fond, l'autre qui souligne tous les éléments en relief. Une telle partition est généralement due à l'usage de différents matériaux de revêtement: à Rome, dans la plupart des cas, on trouve la brique pour les plans de fond et le travertin pour les éléments en relief. Très souvent, toutefois, pour raison économiques, ces matériaux de revêtement étaient appliqués seulement à certaines parties de la façade, tandis qu'on cherchait dans les autres parties d'en réproduire la couleur et la conformation par la technique "pauvre" de l'enduit. On peut maintenant affirmer sans aucun doute que la couleur des surfaces architectoniques est la couleur des matériaux desquels elles sont revêtues, ou bien desquels on veut faire paraître qu'elles soient revêtues. Entre autres raisons de cet usage, il y avait surtout l'ostentation d'une apparence de bienséance, symbolisée d'ordinaire avec des matériaux coûteux, comme la pierre, qui devait s'accorder à la nécessité d'économiser sur le travail et sur le prix des matériaux.

Ces considérations nous donnent la possibilité d'identifier quelques prototypes, c'est-à-dire quelques édifices de particulière importance monumentale qui sont devenus points de repère et modèles quant à l'usage du revêtement, et donc des couleurs.

Un édifice qui, de ce point de vue, a sûrement influencé l'architecture suivante est le Palazzo Riario ou della Cancelleria, dont la façade principale est entièrement revêtue avec des plaques de travertin.

Le Palazzo Massimo alle Colonne a un revêtement dérivant de celui de la Cancelleria. Mais dans le premier, au lieu des tables en travertin de la bande de soubassement, il y a des tables saillantes, réalisées avec un enduit imitant et continuant le travertin; au contraire ce dernier est employé encore dans les bandeaux à cartouche des fenêtres.

Le revêtement de la façade du Palazzo Lancellotti à Piazza Navona dérive du même modèle: dans ce cas aussi on a employé des tables d'enduit imitant le travertin. Le matériau pierreux, au contraire, est employé seulement dans l'appareil à bossages angulaires, dans les cordons et dans les bandeaux des fenêtres et du portail. La coloration actuelle de l'édifice, puisqu'on peut facilement discerner le ton du travertin authentique du ton du faux travertin, fait ressortir, paradoxalement, juste ce que l'on voulait dissimuler: l'emploi d'un matériau "pauvre", l'enduit à chaux, à côté d'un matériau noble, la pierre.

Le rapprochement entre les paraments en briques reguliers et visibles pour les plans de fond et les reliefs architectoniques en travertin trouve dans le Palazzo Farnese et dans les Palazzi Capitolini une réalisation complète. Cette combinaison sera jusqu'à nos jours une des plus diffuses dans l'architecture romaine et non pas seulement romaine. Il est possible d'en indiquer une liste très variée: la Farnesina ai Baullari, le Palazzo Baldassini, l'Oratoire des Filippini malgré la caracteristique articulation borrominienne, l'escalier de Trinità dei Monti, la façade de la Galleria Colonna.

Dans plusieurs cas, toujours afin d'économiser, on employait, pour les paraments, des briques aux bords non complètement équarris, des joints irréguliers et non pas serrés; par conséquent il y avait des interstices de mortier découvert, comme on le voit par exemple dans la paroi latérale du transept de S. Andrea delle Fratte. L'exigence d'une régularisation du réseau géometrique des briques est ici réalisée au moyen d'une couche de finissage couleur brique où on a successivement façonné des joints. En d'autres cas les briques sont imitées par une couche d'enduit travaillé, et naturellement du même ton chromatique. La façade de l'Albergo del Sole à Piazza del Pantheon, dans sa partie la plus ancienne, la droite, montre des briques authentiques. La partie gauche, qui à été successivement construite sur le modèle du noyau originel, imite le parement avec une couche d'enduit travaillé. Un exemple de surface à briques parfaitement imitées est celle d'un palais situé au coin entre Via Due Macelli et Via Capo le Case. Souvent l'enduit travaillé à briques cache et régularise une maçonnerie en briques, comme on le voit dans un détail du Palazzo della Sapienza.

Un autre exemple d'une maçonnerie en briques imitées peut être observé dans la série d'édifices projetés par Valadier à Piazza del Popolo où non seulement les briques mais aussi les bossages rustiques du soubassement sont simulés avec un enduit imitant le travertin.

Le palazzo Sora, à Corso Vittorio Emanuele, bien qu'en grande partie modifié, est un résumé des variantes de la combinaison travertin - briques. Sa partie inférieure, qui est plus ancienne, montre toutes les saillies en travertin, les plans de fond ont des briques à vue au premier ordre et des imitations de brique au deuxième.

Dans l'élevation plus récente l'enduit imite le travertin dans les saillies (aujourd'hui erronément colorées en jaune), tandis que les briques sont imitées simplement avec la couleur.

Dans les façades des églises on a généralement une préponderance de l'emploi de matériaux nobles, et à Rome de l'habituel travertin. On trouve déjà des revêtements de ce type dans l'église de S. Agostino (XV siècle); plus tard, avec l'église del Gesù, ils s'étendront à presque tous les édifices ecclésiastiques de l'âge de la Contre-réforme. Mais il y a aussi, pour raisons d'économie, quelques exemples de travertin imité. L'imitation est particulièrment évidente dans l'église del Santo Nome di Maria (XVIII siècle), vis-à-vis de la Colonna Traiana, où l'emploi du parament pierreux se limite au socle, au soubassement des couronnement. Le dernier ravalement de l'église a altéré la continuité des éléments architectoniques. Ce détail du soubassement montre un résidu du vieil enduit de revêtement qui simule les claveaux de travertin par une imitation des valeurs formelles et tonales.

A Rome on trouve, surtout au cours de XVIII siècle, des exceptions à la règle des couleurs imitant les matériaux. A côté des saillies, toujours en travertin (vrai ou faux), les plans de fond offrent souvent des tonalité très claires, de la gamme gris-bleu ciel. Cela peut être démontré par le détail d'une des vues par Van Wittel et confirmé par la vue du Quirinale. On a déjà trouvé une confirmation dans les documents d' archives, qui indiquent la "couleur de l'air", le "palombino" et le "gridellino" (de l'expression française "gris de lin") comme des tons habituels dans beaucoup d'édifices à Rome.

La polychromie des édifices supposait la possibilité d'y effectuer des peintures à fresque. Mais les exemplesqui demeurent sont peu nombreux. A Rome on en peut indiquer trois typologies: la grisaille de Palazzo Pirro, les fresques sur la façade d'un petit palais à Piazza S. Eustacchio, les "graffiti" sur un édifice de Tor di Nona.

Les exemples jusqu'ici illustrés nous permettent de comprendre combien il est rare de trouver de restaurations d'édifices qui interprètent correctement les intentions de l'auteur du projet et qui respectent les rapports chromatiques suggérés par les matériaux de revêtement; il s'ensuit souvent qu'on brise la continuité des éléments architectoniques ou que l'on nie leur articulation. La récente restauration du Palazzo Muti Bussi, vis-à-vis de l'Ara Coeli, montre qu'on n'a pas compris la valeur de l'appareil à bossages angulaires et de tous les reliefs du premier, du deuxième et du troisième étages, qui naturellement auraient dû être de couleur travertin. Les restaurations du palais de l'Ambassade à Piazza di Spagna, du Palazzo del Gallo di Rocca Giovine à Piazza Farnese, du palais de la Sip à Corso Vittorio Emanuele, de l'Hôpital du Celio, parmi un gran nombre d'autres exemples sont la démonstration d'une telle incomprehénsion.

Une solution également erronée et contradictoire par rapport à la précédente, nous est montrée dans la restauration de palazzo Vidoni-Caffarelli, dans la partie supérieure duquel les demi-colonnes et tous les autres reliefs résultent aplatis par une teinte jaunâtre très semblable à celle des plans de fond. De même façon, dans le Palazzo Bonaparte la subtile et précieuse définition des surfaces en travertin, qui sont, de plus, modulées sur la base de différents techniques de travail, à été éteinte et anéantie dans une monochromie indifférenciée, qui assimile les reliefs pierreux aux plans de fond, qui originellement avaient une aile de briques à vue.

L'intervention encore en cour sur la scénographie conçue par Valadier au Pincio risque, à ce que l'on peut voir à présent, d'altérer les rapports chromatiques originels, basés sur la bichromie travertin - brique.

Les problème qu'on a traité ici, est de façon notoire ni semple à poser, ni rapide ou facile à résoudre. Le présent exposé est une contribution raisonné pour une correcte appréciation du signifié de la couleur dans les choix opératives des interventions de restauration architectonique. Ces choix doivent en tout cas supposer une évaluation historique et technique précise. Aujourd'hui pour chaque édifice qui vient d'être couvert par les échafaudages des chantiers, on doit formuler, sur le résultat final, un point d'interrogation.

Des travaux étant en cours, la présente recherche n'est pas encore terminée.

MORTAR STUDY IN FINLAND.
MAINTENANCE OF HISTORIC BUILDINGS

Thorborg Perander *

SUMMARY

Jointing mortar in old Finnish fortifications from the 14th century has been very durable up to the present day. An effort has been made to imitate such mortar by adding to ordinary lime mortar calcinated clay.

The test results of two different laboratory tests have, however, been contradictory; on the one side good results and on the other side very poor results.

Investigations are going on to find suitable joint- and plaster mortars for the maintenance and repair of old buildings in Finland.

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Background

Old historical buildings made of natural stone and bricks need continuous maintenance. Jointing mortar shall be renewed and the rendered surfaces shall be rendered because of continuous deterioration.

However, the problem is that we have not in Finland found such mortar that could withstand all the stresses it is subjected to in an old brick wall. The base is very heterogeneous: natural stone, old mortar that can be very deteriorated, or base of bricks with varying jointing mortar. Tests have been made with several different mortars, lime mortar and lime cement mortar, but none has so far proved good enough.

The quality of mortar in old historical buildings

Some years ago about twenty plaster and jointing mortars from different old fortifications and other historical buildings were investigated. The aim of the investigation was to find out the composition and properties of the mortar used in Finland from the 14th to the 19th century. These investigations deal primarily with the binder/ballast content and particularly with the binder types.

The mineralogical composition of the binders was investigated with x-ray diffractometer and this structure was studied with scanning electron microscope and analyser. The studies showed that the old mortars contain silicate products, and it was assumed that they partly originated in the crushed brick mixed with the mortar and possibly partly in the products of the reaction between the reactive components (pozzolanas) and calcium hydroxide in the mortar. These silicate products can contribute to the good durability of the mortar.

Laboratory tests on mortar containing calcinated clay

As it was possible that crushed brick could form silicate products in the mortar, we were interested in testing the phenomenon in the Laboratory. Prisms of mortar were made of lime, calcinated clay, i.e. by firing at 700°C, and sand.

The compressive strength of the prisms was higher when kept in 95 % RH than when kept in 70 % RH (70...75 % RH is the most suitable humidity for lime plaster). In addition, the compressive strength in 95 % RH was about three times higher than for lime mortar. These results could indicate that the calcinated clay showed latend pozzolanic properties.

More comprehensive laboratory tests were made on some different mortars including two that contained different quantities of calcinated clay. Besides compressive strength, also the water absorption properties, porosity, drying shrinkage and resistance to varying frost, rain and heat, were studied.

The results of these investigations were, however, not expected, because the mortar containing calcinated clay was not better than lime mortar, and it was much weaker than the lime-cement mortar. Shrinkage was very strong, which was visible from several cracks formed already during the first hours of the drying phase. The mortar did not stand frost and rain at all, but the LC 50/50 mortar withstood them superbly.

It has yet not been clarified why the mortar containing calcinated clay did not in the latter case give positive results, but one of the causes may be that the clay used in the more comprehensive tests was not of the same type that the clay in the preliminary tests. So far it has not been studied whether the clays in special tests show pozzolanic properties after calcination at 700°C.

Research in 1981 - 1984

An extensive project has been started this year on the development of suitable jointing mortars and plasters for maintenance and repair of old historical buildings. The project is financed by the Academy of Finland and the Technical Research Centre of Finland, where it will be carried out.

The goals of the project are

1) an inventory of the condition of plasters and mortars in the old historical buildings in Finland
2) to gather information from literature and directly from various research institutes and laboratories in Europe
3) to study the chemical composition of the old mortars and their physical properites, with special emphasis on the pore structure and distribution, with optical methods
4) to develop one or several mortars that correspond to the requirements that the old buildings of natural stone and bricks impose on jointing mortar and plaster
5) field tests with the developed jointing mortars and plasters and to draft a proposal for repair methods.

The project includes also a study of brick deterioration in old buildings, how advanced the deterioration is, and how it can be controlled.

The project is expected to be finished at the end of 1984.

Section 2 :

Grouts and injections in ancient masonry.

Coulis et injections dans les maçonneries anciennes.

GROUTING FOR STRENGTHENING THE FOUNDATION OF THE QUTB MINAR, DELHI

R. SENGUPTA*

SUMMARY

What the leaning tower is to Pisa, the Qutb Minar is to Delhi, an ancient city and the capital of India which is identified by the landmark. The problem both the towers pose also pertains to the foundation. The original foundation with cavities is considered to be insufficient for the safety of the structure of the Qutb Minar. In this paper the author discusses the various investigations conducted to find out the condition of the materials vis-a-vis the structure and the grouting operations executed to strengthen the foundation of the minar.

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24 August, 1981

INTRODUCTION

The stricken look with fractured stones on the facade of the Qutb Minar caused concern to its custodians if all was well with the landmark of Delhi. Like the contemporaneous (12th - 14th century) tower of Pisa, a companile to a cathedral, the Minar is also an adjunct to a mosque but its lean is much less to make any comparison between the two worthwhile. The tilt in the Qutb Minar is 2'-1" whereas the tower of Pisa leans 16'-6" away from the perpendicular. Nonetheless, the problem they pose is the same as strengthening the foundation.

From the following account of repairs, it will be seen that the Qutb Minar has been receiving as much attention in the recent past as it did during the mediaval period. Consequent to the damages caused by a severe earthquake shock in 1803, major structural repairs were carried out between 1805 and 1828. The cupola put up during the repairs, in replacement of the fallen one, was taken down in 1848 as its design was based on conjecture and looked incongruous on top of the minar. Bulges in the masonry of the minar and cracks appearing in the veneering stones were noted earlier with concern. About the likely causes of their development it was recorded: 'It is not improbable that the displacement of the masonry is the result of settlement that occurred while the tower was being built, due to the work being "run up" too quickly and to the excessive use of the mortar'. After the repairs carried out in 1920, the minar was again examined in the early forties and major structural repairs were executed in 1944-49. An account describes the nature of works undertaken: 'The veneer in general at the Qutb Minar and particularly the inscriptional bands had bulged out badly at places and started falling down. A few twists and bulges had also appeared on the shaft. The affected regions were carefully reset in best mortar after securing them firmly with dowels of stainless-steel encased in cement mortar. Prior to this, cracks in the hearting were grouted and made water-tight'. In 1960 and thereafter in 1964 the successive officers in-charge of the monument expressed their concern over the development of cracks in the stones on the outer face of the minar and suggested for its thorough examination. The foundation of the minar was strengthened by grouting (in 1971-72) on finding its unsatisfactory condition after conducting necessary examinations (in 1964-70) which processes are narrated in this paper.

THE MONUMENT

The Minar

Although construction of the Qutb Minar, as a Tower of Victory and a manar attached to the Quwwat-ul-Islam mosque was started in 1199 by Qutb-ad-Din Aibak, the Turkish commander of Muizz-ad-Din Ghori who ruled from Ghazni, it could be raised by him upto the first storey i.e. only 95 ft. Qutb-Ad-Din's son-in-law and successor Iltutmish added three more storeys and raised the height of the minar. In its present form (fig.) including the addition and alteration effected by Firozshah Tughlaq in the portion beyond the third storey, it is 238 ft. high, about 15 ft. higher than its forerunner at Jam in Afghanistan. As was the practice with the Ghori rulers, the minar at Jam was also erected as a Tower of Victory by Ghiath-ad-Din (1157-1202).

Prior to the construction of the Qutb Minar, though in Afghanistan two minars were erected at Ghazni (11th-12th century), one at Daulatabad (11th century) and one at Siah Posh (10th century) all in brickwork, it is the last one at Siah Posh with which the Qutb Minar resembles in form with semi-circular and angular flutings arranged alternately around its outer face. The angular flutings on the plan of the first three storeys of the Qutb were produced by rotating a square in a circle. Origin of this system may be traced to the Greek method, used in determining the position of steps in the gallery radiating from the platform for orchestra of a theatre, as described by Vitruvius. The angular flutings seen on the fourth storey of the Qutb Minar were obtained by rotating an quilateral triangle which system, according to Vitruvius, was used in Roman theatres. He says: 'The drawings of the plans may be distinguished from each other by this difference, that theatres designed from squares are meant to be used by Greeks, while Roman theatres are designed from equilateral triangles (Morgan, 1960).

Past Repairs

Epigraphic records provide interesting evidences relating to the past repairs executed under the royal patrongage. The Qutb Minar was thrice struck by lightning and repaired successively as mentioned in the inscriptions on its wall. Firozshah Tughlaq restored in 1368, most of the fourth storey and made the minar taller than what it was. Sikandar Shah Lodi's inscription, on the entrance doorway at the ground floor, mentions his carrying out repairs in 1503, but the nature of damage and extent of repairs are not known. Delhi being situated in a seismic zone, it experiences occasional earthquake tremors. The latest in the series was in August 1803 when a shock of considerable magnitude had caused appriciable damages to the minar and its crowning cupola was thrown down.

Masonry used

Firozshah Tughlaq had effected changes in the use of materials in the two top storeys. Red sandstone in the interior casing and marble on the exterior face were introduced. Otherwise, in the construction, the exterior face of the central shaft and the inner face of the minar shell have ashlar facing of Delhi quartizitic stones. The veneering is made of sandstone predominately of red colour though large quantity of stones of buff colour has also been used. The hearting of the shell is made of rubble masonry held together with iron dowels and set in sand and lime mortar. The presence of brick powder in the lime mortar indicates its having been used as an additive.

THE INVESTIGATIONS

Mortar

Appearance of cracks on the veneering stones made the authorities to have a close look at the structure and carry out investigations to find out what was wrong with the minar. Analyses of specimens of mortar (fig. 2) obtained from various places of the minar showed presence of appreciable amounts of water soluble salts, mostly sulphates though chlorides have also been detected. Alumina is present in low percentage which is considered hardly sufficient to impart hydraulic properties to the lime. Repeated solution and crystalization of soluble salts present in the mortar has resulted in the weathering of the masonry. The rusting of iron dowels used in the original construction was an important cause of cracking. The deterioration of mortar and the development of cracks due to expansive forces of rusting dowels allowed ingress of moisture leading to concentration of soluble salts. Possibly with the deterioration of mortar there has also been settlement of the masonry.

State of stones

Examination of specimens of stones from the minar showed that the binding matrix in the sandstone had deteriorated. Even in the quartzite and quartzitic sandstone the interlocking grain system had become disturbed and the cementing material leached out.

While any generalization about the deterioration was not possible, in view of the fact that the samples examined were limited in number and had been extracted mostly from the affected areas, the reports supported the hypothesis that the extent of deterioration on stone and mortar was serious enough to affect the overall quality of masonry and its performance.

Verticality

A check in the verticality of the minar was carried out through the Survey of India and a tilt of 25" (fig.) on the south-west was detected. It was observed that for a base of 47 ft. diameter and 238 ft. height, the tilt was not serious. Since no previous record on the verticality of the Qutb Minar was available, it was not possible to say if the tilt noted was of recent origin. It was difficult to establish the time element, because the historical records show that the minar was built in stages and subsequently when the fourth and fifth storeys were re-built or added by Firozshah Tughlaq, the tilt apparently was adjusted to some extent as suggested by the offset of the central shaft at the level of reconstruction.

The Foundation

The minar directly rests on an ashlar masonry pedastal 6'-6" high and roughly 54 ft. square. The masonry immediately below the minar has sunk producing an inward slope 1:4 in the south-western direction and 1:8 in the eastern direction. The differential sinking is in consonance with the direction of the tilt of the minar. The joints in the masonry of the pedastal have been found to be open. Below the pedastal is a platform 4 ft. high which is made of rubble masonry and about 61 ft. square. A trench dug across the minar from east to west shows that the lower footing of the rubble platform rests, on the east side, on masonry set in lime concrete and on the west on a thick rubble packing in mud mortar. On the west side the trench was sunk to a depth of 27 ft. but the natural soil or bottom of the fundation pit was not reached.

The computation of the volume and weight of the minar at each balcony-height showed that the designing of the foundation was not rational. Even assuming that the foundation had initially been designed to carry the load of the first two storeys only, as Qutb-ad-Din Aibak might have intended and the upper storeys being additions, the foundation should have been stronger.

Geophysical work

To find out whether or not the minar directly rests on a rock-bed, the National Geophysical Research Institute was requested to carry out geophysical work at Qutb to obtain the information. Accordingly resistivity measurements (soundings)were taken. Inspite of very limited scope of making adequate measurements, due to presence of various kinds of structures obstructing the layout of lines and distorting current flow etc., the depth of the bed-rock could be located around 150 ft. to 200 ft. In view of the fact that there are exposures of the Delhi quartzite quite close to the minar, the depth seemed to be rather high. But these depths were not inconsistent with the available bore hole data in respect of the two wells in the neighbourhood. Although the depth of the bedrock below the Qutb Minar is likely to be of the same order, one could not be entirely certain due to the rapid variations in bedrock topography. It was, therefore, suggested that a straight or slightly inclined exploratory boreholes should be drilled at the base of the minar for more reliable information in respect of the sub-surface structure.

Core Drilling

Two inclined core holes were drilled into the foundation. The core hole on the south-west of the minar was driven at an angle of 45° to the base line and extended beyond the centre line of the minar to a depth of 35 ft. from the ground level. The core recovered by drilling revealed the strata being hetrogeneous formation (fig. 4) of quartzite stone with medium to fine sand. The matrix of sand with lime seems to be in a loose state. Due to cavities in the masonry, perhaps due to washing out of the matrix, there was loss of water. Frequent caving of the holes indicated that the matrix of sand with lime had not set or was not compact. The other core hole was also driven to the same depth, on the north-east at an angle of 10° to the base line which did not penetrate into the central portion of the foundation. Cores from this whole suggested more or less similar condition of the pedastal and platform. Below these structures rubble stones with earth filling was met. At the bottom, beyond 30 ft. yellowish earth was found.

Observations

Considering the structural stability, it was felt:

(a) the weakness of the foundation could be the cause of the tilt which though within safe limits, was liable to increase;

(b) the differential settlement appeared to be chronologically the primary reason for the development of some of the cracks, though subsequently other factors might have also contributed towards it; and

(c) the stress in the masonry as calculated, namely, 7 tons per square foot approximately, was within the permissible limits, but left little margin of safety. At the same time, there was no doubt that there had been local stress due to localized deterioration of materials.

Recommendation

In view of the existence of a larger number of pockets in the masonry of the foundation, it was decided that attempt should be made to strengthen the foundation by filling the voids both below and around the minar proper with cement grout, keeping the grout pressure low enough to avoid any up-setting the stress equilibrium already attained. The grout would help to even out the static load of the superstructure as also any excessive stress and reactions in the sub-structure due to incremental load during earthquakes. Consolidation by grouting would also prevent any ingress of moisture or movement of soluble salt into the foundation.

THE GROUTING SCHEME

Phasing of grouting

The grouting operations were planned into phases on and from the 54 ft. square platform (fig. 5A) to the minar base only to a depth of 32 ft. The first phase was further divided into three groups: to begin with the holes along the periphery of the platform and 1'-9" away from the outer edge were to be grouted to create a curtain to prevent any mortar to run out of the basal area; in the second group the holes around the minar were to be grouted; and in the third group the holes in the four corners would be grouted.

In the second phase the sub-structure of the foundation would be grouted through holes bored radially (fig.). As on the north and south there are structures which created obstructions, grouting was decided to be done from the eastern and western sides through the ashlar facing of the platform (fig. 6B). The minimum distance between two holes at the face was to be 20" and each hole would be taken beyond the north-south centre line to a distance of 5 ft. to allow over-lapping and maximum penetration of grout in the central portion.

The maximum distance between to holes was not to exceed 5ft. in the grid.

Specification of Grout

The grout should comprise of ordinary portland cement (complying with IS 269) and water - clean and free from deleterious substances. Grouting any of the holes would commence with grout with a water-cement ratio (by volume) of 4:1 progressively thickened to 1:1 and again progressively thinned till the stage of refusal. Refusal should be deemed to have been reached when the grout intake is reduced to less than one cft. of grout mixture in 20 minutes.

Pressure

The grout holes (1.3/8" dia) would be drilled with standard non-coring rotary equipment except where core-drilling was called for. A continuous circulation of grout would be ensured and permit accurate pressure control. The pressure would be: the top most 10 ft. of the hole @ 5 psi at the gauge; the second 10 ft. of the hole @ 10 psi at the gauge, and the third 10 ft. of the hole @ 15 psi at the gauge.

Drilling and grouting the holes, however, would be done in stages: for holes inclined not more than 10° to the vertical @ 10 ft. and for holes more than 10° to the vertical @ 5ft. In packer grouting, grouting should be completed in stages from the bottom-most stage upwards in the hole, except the top-most stage which should be grouted without a packer. The stages of packer grouting would be governed by the condition of the grout holes.

GROUTING OPERATIONS

Four upheaval gauges with sensitivity of 0.001" were installed at the four corners to watch if there was any disturbance caused due to the pressure grouting. There was no incident of use of excess pressure to show upheaval at any point. Although at a later stage, when grouting was almost completed and the interior spaces were packed up, grout was seen to ooze out through joints of masonry of the superstructure.

During the grouting operations (fig. 7) it was observed that in areas where the overburden was loose, casing pipes (2" dia.) had to be used.

As was planned, grouting was started from the periphery of the 54 ft. platform (S II) to create a barrier around the foundation so that grout injected inside the area might not escape out or be wasted. In this series there were in all 48 holes with 12 on each side. The maximum quantity of grout consumed in a hole was 251 bags (of 1:25 cft.) of cement in the proportions of 4:1 to 1:1 under a pressure of 5 psi; in this hole 237 bags were injected in the first stage. The minimum intake was 6½ bags of cement in 4:1 to 2:1 ratio under a pressure of 5 to 10 psi. Around the minar in the series S I the maximum quantity used was 134½ bags of cement in 4:1 to 1:1 at 5 to 15 psi.

On the whole, consumption of the grout in the interior did not show a very different picture from what was expected on the basis of information gathered by core recovery. Test holes drilled showed that grout did not reach the upper reaches of the 47 ft. diameter base of the structure of the minar. Horizontal holes were, therefore, drilled from east to west and vice - versa to a depth of 22ft. in two stages and grouted to fill in the voids.

CONCLUSION

The information gathered regarding the foundation of the Qutb Minar has set at rest all speculations about its nature and form. Grouting, on the other hand, has been able to consolidate the loose fabric of the masonry of the foundation and to distribute the load over a wider area.

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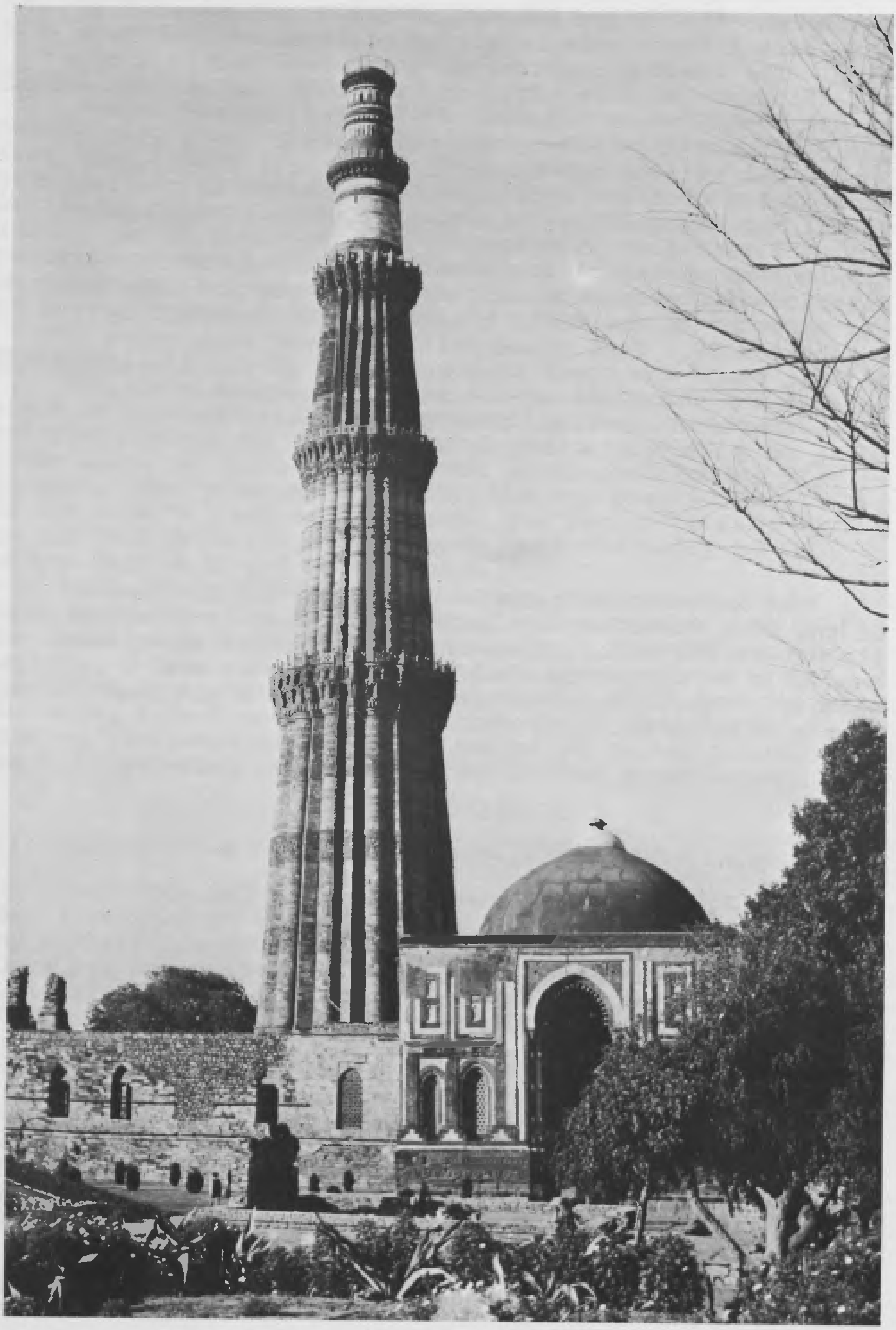

Fig. 1 - The Qutb Minar, Delhi

Fig. 2 **ANALYSIS OF MORTAR SPECIMENS FROM THE QUTB MINAR**

Sl. No.	Description of specimens.	Insoluble residue (In Hydrochloric acid.)	Fe203	Al 203	Cao	Mgo	Loss on ignition.	H20	Water soluble salts.	CO2
1	2	3	4	5	6	7	8	9	10	11
1.	Mortar from first storey - outer face.	62.6	1.11	4.23	16.51	0.25	11.27	2.02	1.69	Not determined
2.	Mortar from second storey - outer face.	52.98	0.86	2.24	23.5	1.56	14.56	0.70	0.24	-do-
3.	Mortar from second storey-inner face	61.53	1.66	2.89	17.76	0.66	14.39	1.5	1.7	12.92
4.	Mortar from third storey-inner face	72.68	1.61	2.00	9.46	0.85	7.1	3.37	3.33	Not determined.

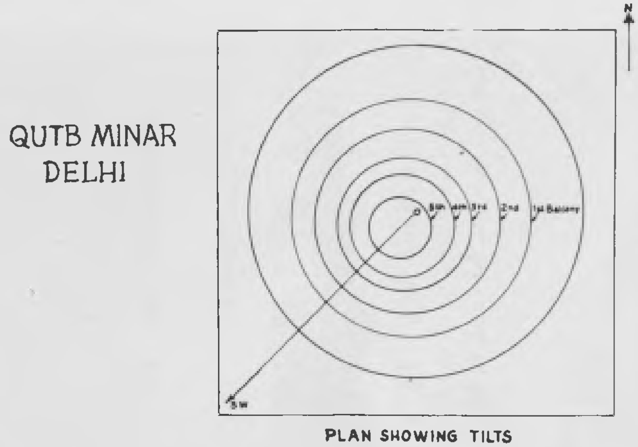

Fig 3a - The Qutb Minar: Plan showing the tilts

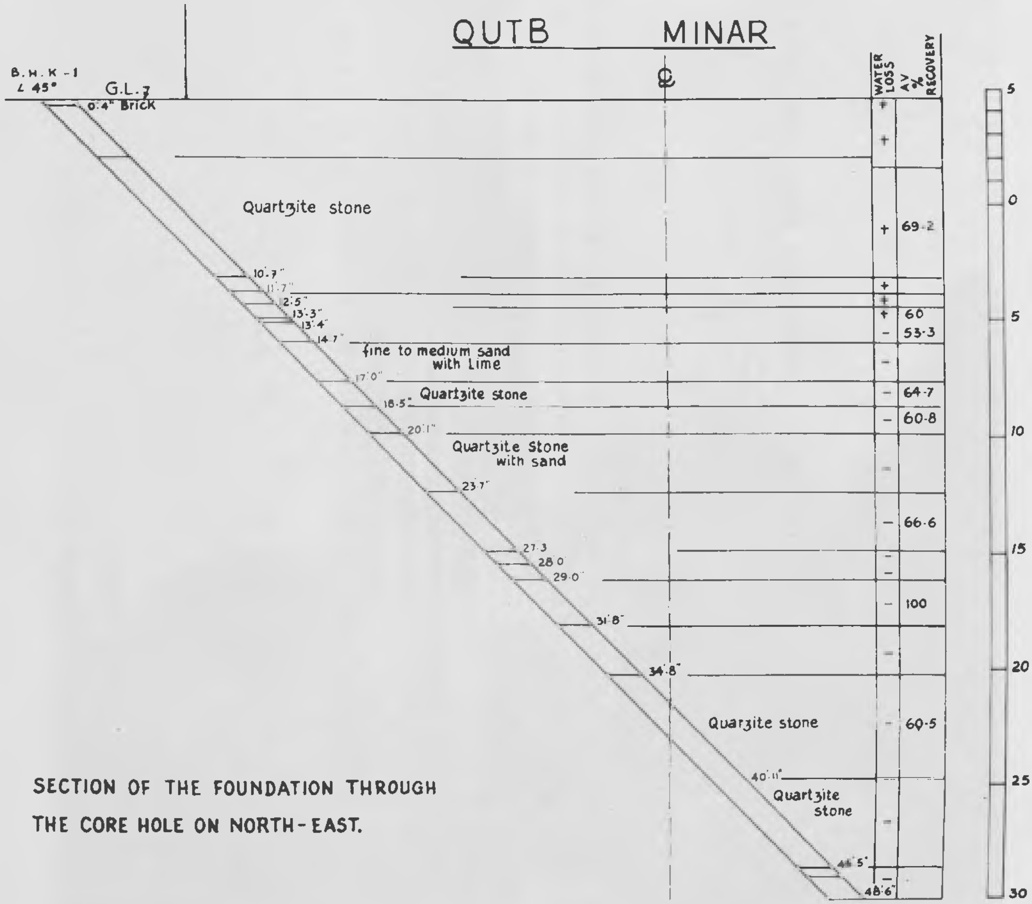

Fig. 4 - The Qutb Minar: Section of the foundation as renealed by core hole bored on the north-east

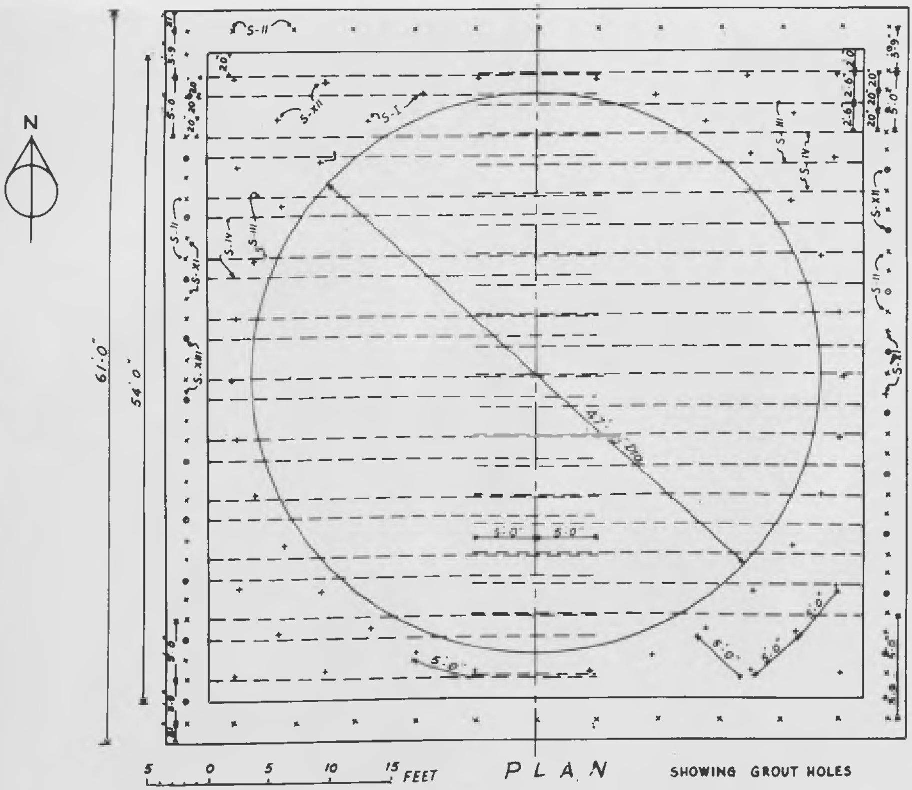

Fig. 5a - The Qutb Minar: Plan of square platform showing arrangements of grout holes

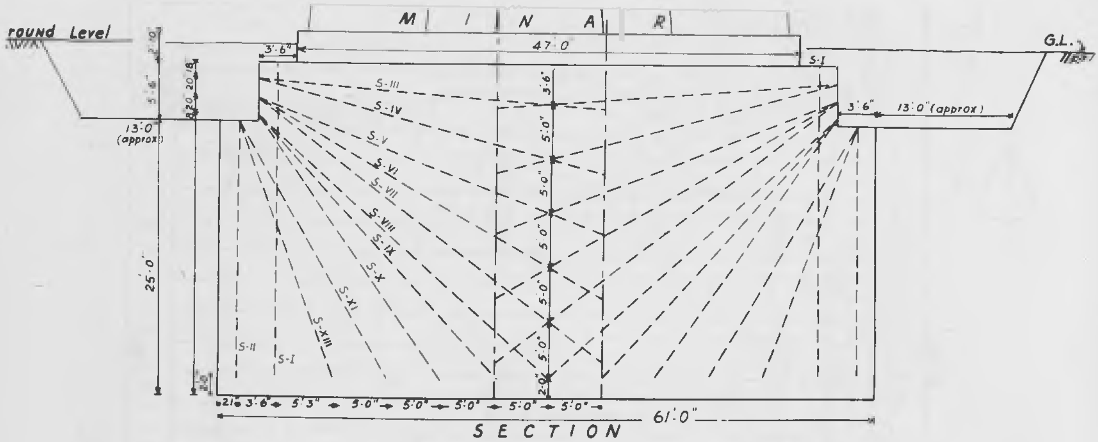

Fig. 6a - The Qutb Minar: Section of the foundation showing radial grout holes

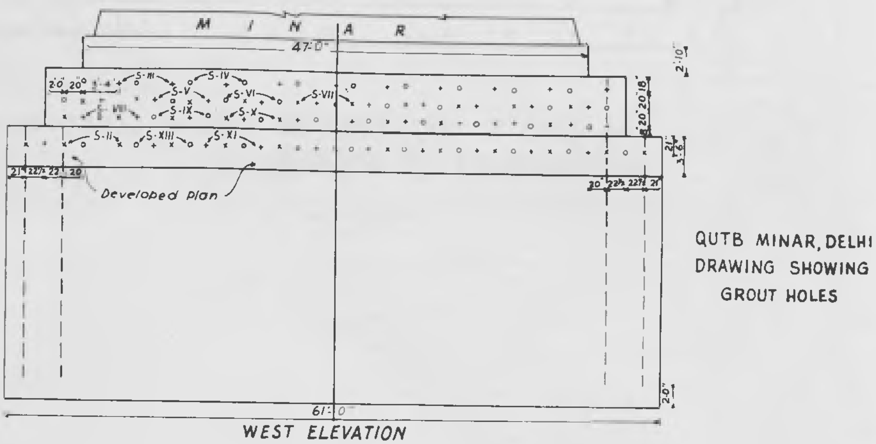

Fig. 6b - The Qutb Minar: Section of the foundation showing arrangement of grout holes on ashler facing of the plinth

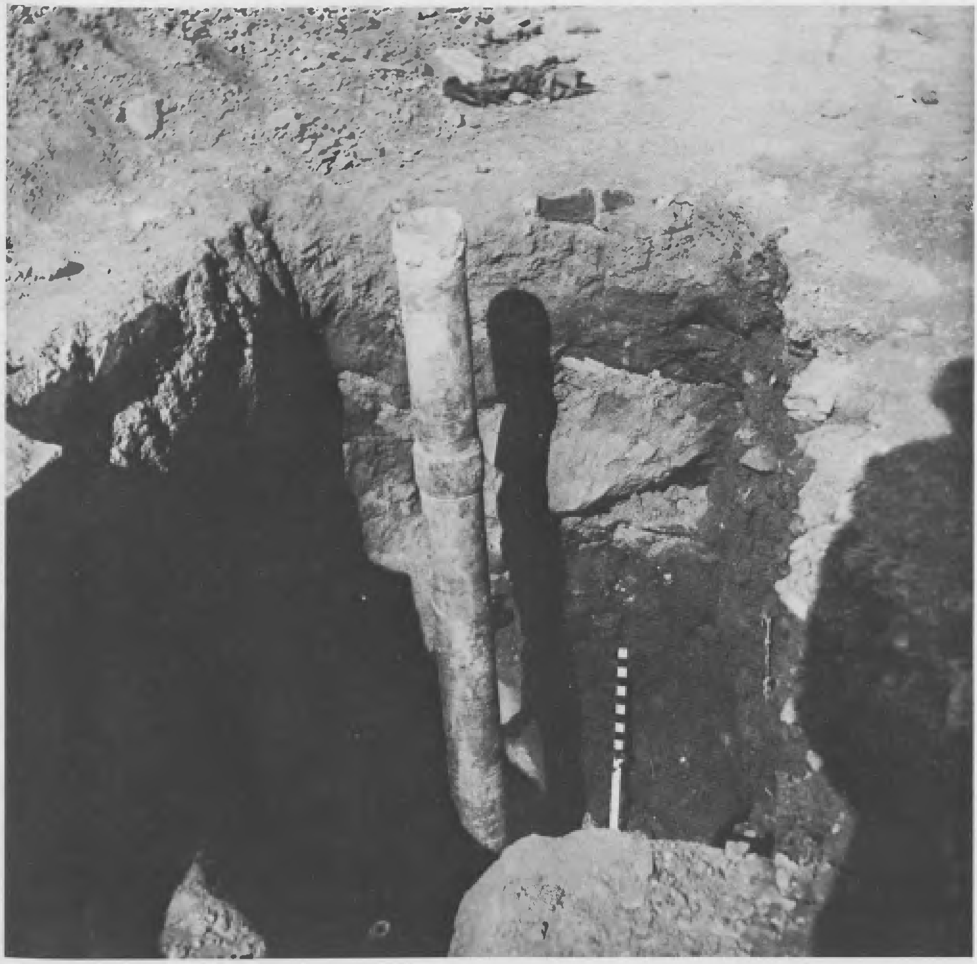

Fig. 7 - The Qutb Minar: A casing pipe used in grouting where the overburden was of loose material

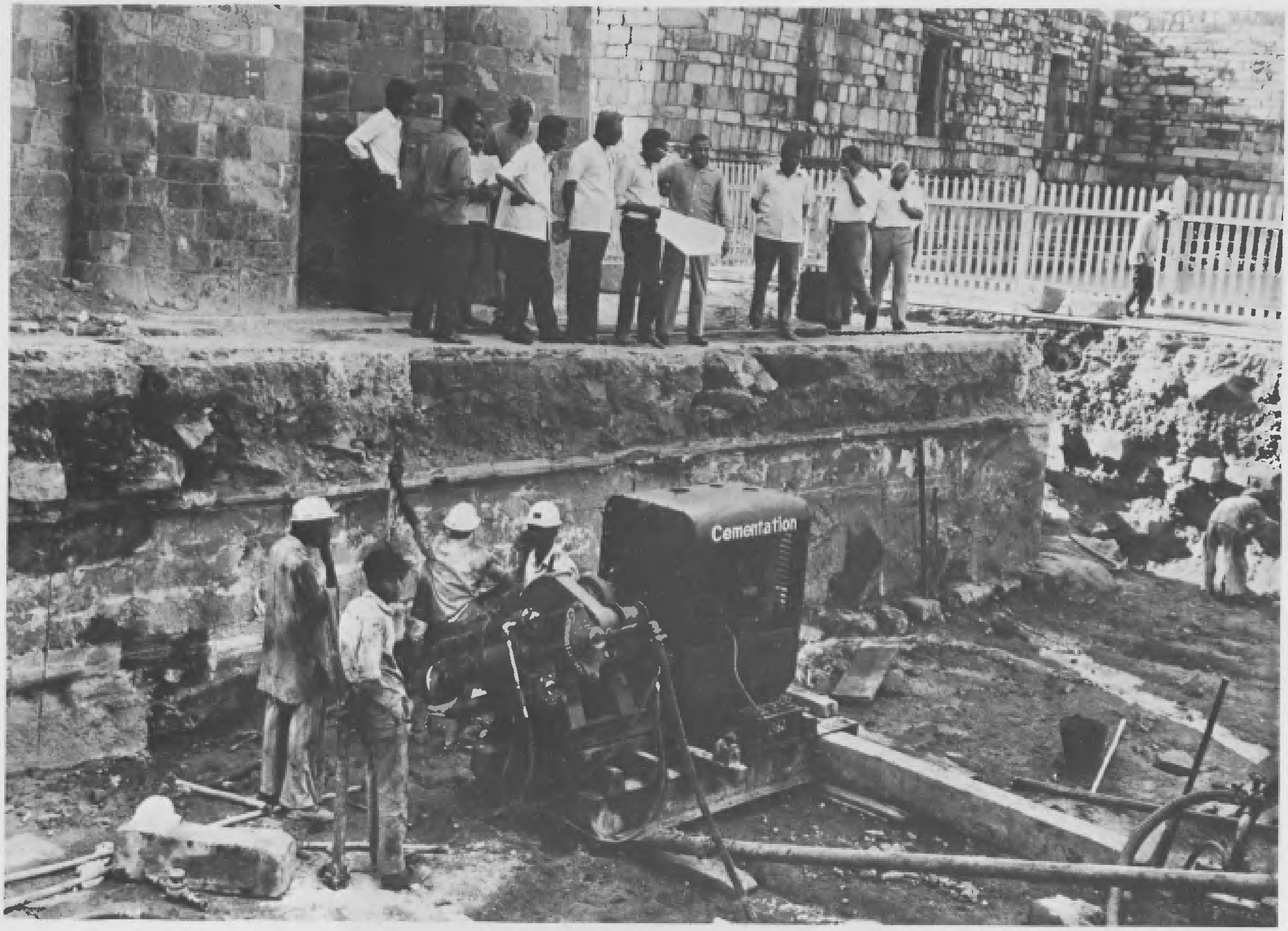

Fig. 8 - The Qutb Minar: Grouting from east through inclined holes

Fig. 9 - The Qutb Minar: Grouting holes around the Minar

RENFORCEMENT DES MACONNERIES PAR INJECTIONS DE COULIS DANS LA REGION NORD-EST DE LA FRANCE

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(C.E.B.T.P.)

RESUME

Etude concrète de la consolidation d'une maçonnerie d'Eglise du XIIIème composée de gaize (pierre argilo-calcaire friable) et hourdée à la terre corroyée.

Des coulis à base de silicate de soude et de bentonite-ciment ont été employés respectivement pour consolider le mortier à la terre et pour renforcer la maçonnerie.

Des mesures comparatives des caractéristiques mécaniques de la maçonnerie réalisées avant et après injection, ont permis de contrôler l'action de ce confortement.

La mesure de la vitesse de propagation du son par transparence, ainsi que des prélèvements de maçonnerie, par carottage, mettent en évidence l'évolution de ces qualités.

Nous avons observé que le coulis de bentonite-ciment n'obture que les vides supérieurs au demi-millimètre et ne diffuse pas dans le mortier de terre qui, par contre, est imprégné par le coulis de silicate de soude.

PRELIMINAIRES

I-I De tous temps lorsque les pierres calcaires furent employées dans la construction, il est apparu que les ouvrages anciens se dégradaient par l'action, souvent conjuguée :

- de l'eau infiltrée et du gel qui délitent les matériaux,
- des forces diffuses dues à des tassements et déversements des maçonneries, parfois conséquences d'efforts extérieurs qui déliaisonnent les massifs et les murs, ces forces engendrant des fissures apparentes ou internes susceptibles de provoquer, immédiatement ou à terme, leur effondrement.

I-2 Des remèdes aux désordres dus aux effets de l'eau peuvent être appliqués par un meilleur choix des matériaux et une étude plus poussée de leur mise en oeuvre, suite aux travaux du service des Monuments Historiques, du Laboratoire de Recherches des Monuments Historiques (L.R.M.H.).

Par contre, les phénomènes qui causent le déliaisonnement des maçonneries né paraissent simples qu'en théorie. Aujourd'hui, leurs traitements complexes ainsi que les résultats des essais effectués, sont peu connus

L'objet de cette étude, avant toute pratique, est de noter les remarques à l'occasion de travaux de restauration de maçonneries dans les régions NE de la France, régions où des matériaux presque exclusivement calcaires ont été employés pour la construction et les réparations des monuments anciens.

Elle comporte dans une seconde partie le compte rendu d'une recherche technique en laboratoire avec expériences in situ suivi d'un projet de traitement des maçonneries, fonction de la diversité des cas d'espèce, présentés par chaque chantier de restauration ; cette partie a été rédigée avec la collaboration du CEBTP, Service d'Etude des Matériaux (1).

II. LES MATERIAUX EN OEUVRE

II.1. Selon les monuments, en pratiquant les travaux de maçonnerie, le restaurateur rencontre des murs ou massifs constitués de matériaux divers, diversement mis en oeuvre, pierre de taille ou moellons, extraits des carrières locales ou régionales.

II.2. Schématiquement, ils se classent suivant 3 types :

- les maçonneries d'appareil régulier, dont les pierres sont débitées et taillées géométriquement et soigneusement. Les lits règnent horizontalement même si les blocs ne sont pas de longueur constante.
- les maçonneries d'appareil moyen, les arètes, chaînes, arases et points porteurs y sont réalisés en pierre de taille, le reste en moellons plus ou moins grossièrement taillés posés en parement avec blocage intermédiaire.
- enfin, les maçonneries faites de matériaux dégrossis, moellons posés en parement, réunis par blocage de petits matériaux, systèmes utilisés le plus souvent pour des constructions économiques.

III. QUALITES DES PIERRES DE CONSTRUCTION

III.1. Les ouvrages de maçonnerie de la zone NE de la France sont exécutés avec des matériaux locaux. Les couches successives de la "cuvette" géologique constituant le Bassin Parisien offrent de bas en haut :

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(1) La Direction Rég. des A.C. de Champagne, les Professionnels du Bâtiment, les Entreprises, J. NOEL, HORY, CHATIGNOUX ont participé par leur contribution et leur travail à la mise en place et à la poursuite des essais ; c'est pour moi un agréable devoir de le faire savoir et de leur dire en même temps ma gratitude pour avoir ainsi contribué à rendre moins aléatoire un travail délicat particulièrement utile aujourd'hui à la restauration des Monuments Anciens.

- Les schistes des Ardennes et les grès de l'Est,
- Les calcaires marneux employés en Meuse, avec les calcaires oolithiques (Bajociens et Bathoniens) gris à Euville de belle couleur jaune en Ardennes, vers Metz et dans la région de Savonnières en Perthois,
- Les calcaires corraliens de l'Yonne et du Jura,
- Les nombreuses couches de craies, craie verte marneuse, blanche et jaune de la Champagne,
- Les calcaires éocènes des environs de Paris,
- Enfin, les meulières de la Brie et de la Brie Champenoise.

III.2. Parmi ces matériaux il en est de très perméables, d'autres moins, des craies gélives et des schistes et meulières à peu près insensibles au gel puisque imperméables.
Aujourd'hui la plupart des carrières répertoriées sont fermées et non réexploitables pour en tirer les faibles quantités de matériaux nécessaires aux restaurations. Pour les travaux actuels des M.H. dans cette zone il faut se fournir dans des carrières encore exploitées fournissant des matériaux dont les caractères géologiques sont compatibles avec ceux des matériaux d'origine.

IV. LES MORTIERS ENDUITS ET JOINTS

IV.1 .L'examen des mortiers réserve quelquefois des surprises.
En restaurant un mur ancien, on rencontre parfois un "mortier" de couleur légèrement plus claire ou plus foncée, il indique souvent une reprise partielle de maçonnerie que l'examen du parement n'avait pas laissé voir au premier coup d'oeil, lorsque l'érosion de la surface a fait disparaître l'aspect de la taille.
Suivant les époques le mortier diffère, les restaurations les plus récentes sont maçonnées avec un mortier généralement plus dur que l'ancien et comportant une plus grande quantité de chaux et de sable, au lieu de mortier de terre.

IV.2. D'une façon générale le mortier de terre a été employé en toutes régions depuis l'époque gauloise et peut être plus tôt, jusqu'au XIXème. La diffusion des résultats des expériences de Vicat et de ses continuateurs, puis la fabrication et la commercialisation

industrielle de la chaux et du ciment permirent la mise en oeuvre de matériaux offrant une résistance considérable.
Cette règle souffre sans doute des exceptions ! les monuments publics romains étaient construits avec un mortier renommé qui a défié les siècles et le défaut d'entretien. Le moyen âge a connu aussi des mortiers d'une solidité comparable lorsqu'il s'agissait de construire un château d'une grande importance stratégique. L'époque classique de même, ainsi que pour certains grands monuments ou abbayes.
Partout ailleurs, c'est de la terre corroyée, ou de préférence de la boue ramassée sur les chemins mélangée de petits cailloux formant agrégats, petits blocs de craie, graviers calcaires, sables de rivière ou de carrière.

IV.3. Les enduits extérieurs sont traités avec des mortiers de chaux aérienne et sable, plus résistants que les mortiers de terre utilisés pour la pose, généralement appliqués en deux couches: la première ou accrochage avec des granulats souvent plus gros. Lorsque l'enduit devait comporter une deuxième couche lissée à la truelle, celle-ci était souvent suivie d'une légère couche de chaux pure blanche ou teintée appliquée au pinceau.

IV.4. Les enduits intérieurs étaient préparés de même, ou tout simplement réalisés avec un torchis, terre battue mouillée à laquelle étaient incorporés des poils de vache pour éviter les fissurations au séchage et badigeonnées ensuite à la chaux.

IV.5. Les injections et travaux confortatifs entraînent presque à coup sûr la réparation des enduits et joints, sinon leur réfection.
Les joints étaient toujours faits après coup lorsque les pierres du mur restaient apparentes. Ils étaient garnis avec un mortier fait de chaux et de sable très fin et généralement affleurés.
Des causes diverses concourent à la dégradation des mortiers.
Les rongeurs ou petits animaux qui cherchent abri dans les murs y creusent des cavités qu'ils remplissent ensuite plus ou moins complètement de menue paille ou de brindilles végétales généralement dans les parties basses ou les fondations. Dans les parties hautes se sont les loirs les petits oiseaux ou les insectes qui font de même.

Quand le mur est mouillé par la pluie, les arbustes et le lierre prennent racine dans la terre des mortiers; lorsqu'ils sont coupés ou détruits leurs racines pourrissent laissant des vides.
Quelquefois, dans des murs élevés rapidement ou fondés sans grands soins, les maçons n'ont pas complètement garni les blocages qui comportent jusqu'à 5 % de vide en volume !
Le mortier, ou ce qu'il en subsiste, n'est donc pas un élément homogène, ni réparti également dans les murs, sa résistance aux contraintes de charge sera donc différentes selon les points, et des risques de désordres seront toujours latents.

IV.6.- Autre conséquencede la nature du mortier sur son comportement est sa perméabilité.

- L'humidité amollit le mortier de terre, provoquant des déformations sous des contraintes ponctuelles,(les efforts dus aux voûtes ou arcs par exemple).Par contre, la sécheresse provoque des fissures et la pulvérisation de la terre, d'où parfois, l'incorporation de bourre pour éviter le faïençage des enduits de terre notamment.

Les quantités d'eau absorbées par la base d'un mur au contact du sol sont difficiles à mesurer : par comparaison des poids d'un mortier humide extrait d'un mur puis séché à l'air après une quinzaine de jours, la différence peut être de l'ordre de 10 à 15 % du poids sec.

V. OBJECTIFS DU RESTAURATEUR

V.1 Devant un mur lézardé dont les joints sont creux, les parements gonflés et dont certains éléments tombent ou se laissent enlever à la main quel sera le parti vers lequel se tournera le restaurateur ?

1 - La reconstruction,
2 - Le remaillage avec reconstruction partielle,
3 - Le remaillage limité avec consolidation,
4 - Le remplacement des pierres brisées et la mise en place de coulis au cours du travail,

5 - Le remplacement des seules pierres disparues, le recollage de celles fissurées et la consolidation in situ du mur à condition qu'il ne soit pas trop déversé et devenu instable.

Les trois premières solutions qui étaient fréquemment utilisées jusqu'à la guerre de 1914,sont abandonnées aujourd'hui sauf exceptions sur les chantiers normalement conduits.

V.2 Aujourd'hui, en général les entreprises qualifiées font remailler les maçonneries en montant; le mur est garni de bas en haut par des coulis au seau avant pose des blocs de parement par fichage et coulis.

En cas de démontage on constate après le travail une pénétration satisfaisante, les vides sont garnis mais dans une faible zone périphérique seulement.

Le plus souvent on utilise la chaux au lieu du ciment pour favoriser la perméabilité du mur et éviter en théorie, les dégats dus à un mauvais cheminement de l'humidité à travers le mur et permettre son évaporation. Par ailleurs, il est souhaitable par mesure d'économie de limiter les reprises de pierre. Les blocs de parement écrasés sont seuls refaits, ceux fissurés sont recollés in situ, mais les coulis restent toujours difficiles à bien réaliser avec pour conséquences :

- a : liaison précaire des blocages aux parements ou de l'appareil aux blocages.
- b : impossibilité de couler avec une garantie de pénétration suffisante une barbotine de ciment ou de chaux dosées à 1/1 en poids dans un mur fissuré. Des bouchons se forment et le débourrage est un palliatif insuffisant ; le bouchon axial étant franchi il se forme d'autres bouchons latéraux dans des diverticules invisibles et indébouchables.

  Autre cause d'échec dans les maçonneries de craie très fissurées, l'eau mélangée à la poussière qui recouvre les matériaux semble former un film étanche en surface et le coulis désséché n'adhère plus.

VI. ETUDE THEORIQUE ET EXPERIMENTALE CONFIEE AU C.E.B.T.P.

VI.1 Devant ces causes d'échecs fréquents, se renouvellant sur le chantier de restauration de l'église du Château à Ste-Ménéhould (51) l'ACMH fut amené à étudier un procédé plus efficace. Ce travail de recherches et d'essais fut proposé à M. le Directeur Rég. des A.C. et accepté par lui ; la mise en oeuvre fut confiée au CEBTP avec les objectifs suivants :

- Rechercher un procédé de traitement.
  satisfaisant sur le plan technique, sans contre indications, solide dans le temps, esthétique ou neutre, commode et facile à mettre en oeuvre par toutes entreprises,
  bon marché pour ne pas gréver un budjet limité, les résines étant trop coûteuses, l'usage doit en être limité au parement et au collage.

VI.2 A Ste-Ménéhould ce procédé doit apporter un remède satisfaisant au déliaisonnement des maçonneries notamment celles des murs gouttereaux et à cause de l'urgence de cette consolidation pour l'achèvement de la tranche de travaux en cours restauration des nefs et et transepts.

Construite à partir du XIIIème et modifiée plusieurs fois depuis en employant à toutes époques la gaize, pierre locale, argilo calcaire friable et gélive, cette église présentait des murs déversés de 20 à 30 cm dans la nef, situation à laquelle l'explosion d'une poudrière au XVIIème, n'était sans doute pas étrangère. Les voûtes étaient très fissurées et désorganisées malgré des réparations antérieures. Une consolidation interne de ces maçonneries très désorganisées était nécessaire.

Le CEBTP fut donc dans un premier temps invité à :

- étudier les éléments des murs à partir d'échantillons pris sur place, matière des parements et blocage, mortiers,
- étudier des matières convenables pour constituer des coulis satisfaisants,

- enfin, chercher à mettre au point une méthode d'essai, sondage interventions et conséquences, commode pour tous chantiers même petits de moins de 2 m3 de matière injectée.

Les premières investigations se firent sur place le 11.1.79, le 28.1.80 le CEBTP remettait son rapport sur le renforcement des maçonneries par injection de coulis, rédigé par M.M.MAMILLAN assisté de MM. BOUINEAU, CUENDET, ingénieurs et GARNIER, Technicien, un résumé succinct en est fait ci-après.

VI.3 <u>Résumé de l'étude bibliographique</u> :

La régénération d'un mortier complètement décomposé dont le sable et autres éléments sont restés en place, est très délicate. Le coulis de ciment est arrêté dès son premier contact avec le matériau. Le milieu à injecter possède une perméabilité faible et joue le rôle de filtre envers le coulis. Il se produit un blocage de la suspension, laquelle laisse partir son eau et ne progresse plus dans la masse.

On peut penser que les éléments composant l'émulsion employée sont trop gros pour pénétrer les nodules. Il semble donc nécessaire de recourir à des coulis plus complexes.

- coulis stable, d'argile, ciment,
- résines organiques,
- coulis nobles (résines époxy, polyester,..).

Ces coulis peuvent présenter une fluidité proche de celle de l'eau, car leur angle de cisaillement interne reste très faible. Cette caractéristique permet d'effectuer des injections sous faibles pressions et d'obtenir une meilleure pénétration des éléments à consolider.

Il en résulte que les coulis instables ou barbotines de ciment ou de chaux purs ne sont pas recommandés, car ils se bloquent rapidement et, par là, ne diffusent pas dans le matériau.

La consolidation des maçonneries à faible perméabilité peut être traitée de la même manière que les alluvions sablo-graveleuses.

La difficulté réside dans le fait que, la montée en pression du coulis à injecter risque de provoquer des désordres graves dans la structure déjà désorganisée.

Il est donc nécessaire d'effectuer les injections sous très faible pression et, de contrôler les déformations qui peuvent résulter de

la pression interstitielle créée pendant les travaux.
Un coulis stable est une suspension dans l'eau de grains suffisamment petits pour qu'aucune sédimentation ne se produise pendant l'injection.
Une suspension d'argile colloîdale est le type même de ces coulis. Elle doit être fluide, afin que l'injection soit possible et posséder après injection une rigidité suffisante.
L'adjonction d'une très faible quantité de bentonite au ciment permet de réaliser des coulis injectables, stables, avec une bonne résistance à la compression.
En outre, les coulis liquides tels que les gels durs de silicate de soude et de lignochrome, sont :
- susceptibles d'aller dans tous les vides. La fluidité de ces coulis est très grande, quelques centipoises mais la résistance à la compression est peu élevée. Elle peut cependant atteindre 5 N/mm2 dans certaines conditions d'utilisation,(sur le mortier consolidé)

VI.4 <u>Contrôle de l'état de la maçonnerie à consolider</u> :

La décision d'injecter une maçonnerie est très délicate à prendre. Elle ne peut être prise qu'à la suite d'une étude complète des éléments qui la constituent. Le choix d'un coulis dépend de la nature de la maçonnerie et que son coût soit en rapport avec la valeur architecturale de l'élément.

VI.4.1 <u>Essai non destructifs</u> :

L'auscultation dynamique permet de contrôler l'efficacité de l'injection par des mesures comparatives de vitesse de propagation du son (avant et après consolidation).
Mais il faut noter que dans les maçonneries de blocage ou fourrées, la mesure de la vitesse de propagation du son avant injection est souvent impossible en raison de la très mauvaise compacité du mur.

VI.4.2 <u>Prélèvements d'échantillons</u> :

Un prélèvement intact par carottage est instructif, il renseigne avec précision sur la constitution exacte du mur : épaisseur de parement - grosseur des éléments de blocage - état du mortier d'assemblage, vides, etc...
Lorsque le mortier est totalement désorganisé ou lorsqu'il a été entraîné par des infiltrations d'eau, le prélèvement par carottage n'est plus possible car il n'existe plus aucune liaison entre

les éléments qui constituent la fourrure.
Il est alors possible d'effectuer un prélèvement d'échantillons remaniés par un piochage ponctuel de la maçonnerie (après la pose d'un étai pour neutraliser l'affaiblissement causé à la maçonnerie)
Ces prélèvements intacts ou remaniés permettent de définir un indice des vides de la maçonnerie et du mortier de pose utilisé.Cet indice des vides oriente l'expérimentateur dans le choix des coulis à utiliser.
En effet, il faut parfois recourir à l'usage de plusieurs coulis pour consolider les maçonneries anciennes, afin de colmater successivement les grosses cavités, puis les plus petites, pour terminer par la consolidation du mortier. Cet indice des vides donne également une indication sur les quantités de coulis à utiliser et de faire une estimation du coût de l'opération en matière première.
Les prélèvements de mortier ou de terre corroyée servent à la mise au point du coulis.
La définition de la fluidité du coulis dépend :
1) de la perméabilité du mortier,
2) de la résistance escomptée après consolidation.
L'état de la maçonnerie définit également les pressions d'injection admissibles. Une pression trop forte peut détruire une maçonnerie ancienne par écartement des parements. Une pression trop faible ne permet pas au coulis de s'infiltrer dans les capillaires fins du mortier à consolider.

L'apport d'eau dans un mortier ancien (souvent de la terre corroyée) provoque son gonflement, ce qui peut engendrer des désordres importants supplémentaires. Ce gonflement peut atteindre plusieurs millimètres sur un mur de 1 mètre d'épaisseur.
Un essai de gonflement doit donc être effectué sur les prélèvements de mortier.
Lorsqu'il s'agit d'un mortier de terre corroyée, l'essai peut être effectué sur un échantillon remanié moulé à la densité initiale (essai norme américaine S 20 C).
Il peut également se produire un lavage des matériaux par l'eau composant le coulis. Il se produit alors une variation de l'angle de frottement interne ce qui entraîne un effondrement par tassement de la fourrure.

D'autre part, le mouillage des matériaux peut créer une pression hydrostatique à l'intérieur des murs et provoquer de nouveaux désordres (lézardes, fissures, tassements).

VI.4.3 Etat de l'enduit et des joints de parement :

L'injection ne peut se faire que si l'étanchéité des joints entre pierres est bonne. Lorsque le parement en pierre est recouvert d'un enduit, l'étanchéité de cet enduit doit également être satisfaisante. Dans les cas contraires, il faut avant tout envisager la remise en état de ces 2 points particuliers, sinon l'injection n'est pas possible, car le coulis ne resterait pas dans la maçonnerie.
Il faut également s'assurer qu'il existe une étanchéité à la base du mur, sinon le coulis risque de s'infiltrer dans le sol.
Dans ce cas, il faut alors effectuer une reprise en sous-oeuvre ou bien injecter préalablement le sol sur lequel repose la maçonnerie afin d'obtenir l'étanchéité à la base du mur.
La pression d'injection du coulis dépend de la qualité des joints et de l'enduit. Un enduit, dont l'adhérence est trop faible peut être décollé ou arraché pendant l'injection.

VI.5 Choix du coulis :

L'étude sur les matériaux et sur la maçonnerie étant achevée, le choix du coulis peut maintenant se faire.
Il doit répondre aux impératifs cités précédemment, et, de plus posséder les qualités suivantes :

1) Facilité d'injection :

Le coulis doit rester stable pendant la durée d'injection.
Le temps d'utilisation doit être facilement réglable (cas des gels de silice). Sa fluidité doit rester constante du début à la fin de l'injection (contrôle au cône Marsch).

2) Retrait du coulis :

Le coulis doit effectuer le moins de retrait possible, sinon il laisse des volumes vides dans la maçonnerie. Pour les coulis de ciment et de chaux, la décantation doit être la plus faible possible. Ces coulis ne doivent pas perdre leur eaux en arrivant en contact avec le mortier ce qui produit un blocage.de l'injection.
Ils doivent donc être stabilisés par l'adjonction d'argile ou de bentonite, dans le cas de matériaux poudreux ou de mortiers anciens (terre corroyée très avide d'eau).

3) Durabilité :

Le coulis doit être stable dans le temps et ne pas perdre ses caractéristiques mécaniques sous l'action d'agents extérieurs (délavage à l'eau - retrait excessif - tenue au gel).

Il est pratiquement impossible qu'un coulis présente toutes les qualités énumérées ci-dessus. Le choix se portera donc sur le coulis présentant le meilleur rapport qualité/prix. Pour certains points particuliers, il est bon de négliger ce rapport, afin de faire prédominer la qualité.

Il est à noter que les injections ne sont pas en général, réversibles mais par contre, peuvent être très souvent complétées dans le futur par les améliorations des produits et techniques.

VI.6 Essais de laboratoire sur le mortier de terre corroyée :

Des prélèvements intacts n'ont pas été réalisables, ; aussi, avons-nous travaillé sur des échantillons remaniés ou reconstitués.

VI.6.1 Essais de gonflement du mortier reconstitué :

L'essai de gonflement a été effectué sur deux éprouvettes, constituées de matériaux prélevés sur place, selon la norme américaine (S 20 C). Le matériau est écreté au tamis de 20 mm.Il est mis en place dans des moules CBR, à la densité sèche de 1,35 T/m3.

Un gonflement de 5 pour 1000 est constaté en unidimensionnel. Ce gonflement ne représente plus que 1,7 pour 1000 en tridimensionnel, ce qui est faible après une saturation de 24 heures et ne semble pas devoir présenter de risques de désordres pour la maçonnerie en place qui est très peu compacte. Il faut plutôt craindre un lavage des matériaux, par l'eau et d'autre part, le mouillage des matériaux peut créer une pression hydrostatique à l'intérieur des murs.

VI.6.2 Mesure de la densité apparente du mortier de terre prélevé intact :

Les mesures ont été effectuées sur cinq éprouvettes par pesées hydrostatiques, et ont donné en moyenne une densité sèche apparente de 1,60 T/m3 et une porosité de 40 %.

VI.6.3 Régénération du mortier de la maçonnerie :

Elle ne peut se faire aisément que si l'altération du mortier est telle qu'il existe des passages suffisamment gros pour que le coulis puisse circuler.

Pour ce genre d'injection, il est nécessaire d'effectuer des forages assez rapprochés (quelques dizaines de centimètres). La pression de refus dépend avant tout de l'épaisseur et de la nature de la maçonnerie.

Pratiquement, la régénération du mortier composant la fourrure des murs sera donc possible selon la perméabilité du matériau en place. Le colmatage des cavités sera assuré par un coulis de bentonite-ciment alors que la consolidation des éléments fins devra être faite par injection de coulis plus nobles (silicate de soude, ou résine polyester, etc..). L'étude en laboratoire a consisté en une série de tests vérifiant les possibilités d'injections de ces différents coulis. Dans ce but, il a été réalisé des éprouvettes en matériau reconstitué, lequel provenait de la fourrure des murs.

Réalisation des éprouvettes :

Le matériau est d'abord écrété à 20 mm de Ø ; ainsi les éléments de grosse taille faisant partie du remplissage du mur sont supprimés. Ces derniers ne devant pas être imprégnés ni consolidés, à ce stade de l'étude, nous pouvons ainsi travailler sur des éprouvettes de petites dimensions

Dimensions des éprouvettes : Ø 140 mm.
h 265 mm.

Elles sont réalisées dans des cylindres en altuglass comportant, à une extrémité, une plaque collée, de même nature, et percée en son centre pour permettre l'injection des coulis par un tuyau en polyvinyl, raccordé à un pot de pression.

Le matériau est mis en place par vibration sur une table. La teneur en eau de gâchage est de 23 %. Nous obtenons ainsi une densité sèche moyenne de 1,55 T/m3. Les éprouvettes sont ensuite séchées à l'étuve à 40°C.

Caractéristiques mécaniques d'une éprouvette non injectée :

- Vitesse de propagation des ondes sonores : cette mesure est très délicate et devient imprécise pour le matériau reconstitué. Nous arrivons cependant à établir une valeur proche de 1.300 m/s.
- Résistance à la compression sur une éprouvette de 140 mm de hauteur : 0,34 MPa.

VI.6.4 Injection des éprouvettes de mortier :

L'éprouvette est placée sur un bâti métallique. La plaque supérieure, perforée, reçoit la réaction de la poussée d'injection. Le tube d'altuglass, transparent, permet de voir la remontée du coulis au travers du matériau. L'éprouvette est imprégnée verticalement par remontée chassant ainsi l'air enfermé dans le mortier. Ce principe permet un remplissage optimum.

Silicate de soude :

Nous avons essayé 4 compositions à base de silicate de soude pour finalement retenir la formule suivante :

Silicate de soude 3.3/ 38/40°	48,9 %
Durcisseur 2000	9,9 %
Eau à 20°C	41,2 %

Cette composition donne un coefficient de neutralisation égal à 70 % et une viscosité de 6 centipoises. L'éprouvette est imprégnée sous une pression de 0,12 MPa. Les caractéristiques mécaniques obtenues sont :

Densité sèche	1,61 T/m3
Vitesse de propagation du son	1.520 m/s
Résistance à la compression	1,42 MPa.

Bentonite-ciment :

Nous avons essayé un coulis à base de bentonite-ciment de composition suivante :

- ciment CLK 45	50 kg
- eau	40 kg
- fluidifiant	1 kg
- bentonite hydratée à 1000 %	16,5 kg.

Le mélange est préparé dans un agitateur à haute turbulence afin de bien décoller les grains de ciment les uns des autres.

Le temps d'écoulement au cône Marsh avec un ajustement de 6 mm est de 17 s $<$ t $<$ 19 secondes.

La décantation des éprouvettes (Ø 54 mm h = 200 mm) après 24 h. est de 20 % $<$ D $<$ 23 %.

Nous avons constaté que le coulis ne pénètre pas dans le matériau reconstitué de l'éprouvette à la pression de 1,5 daN/cm2.

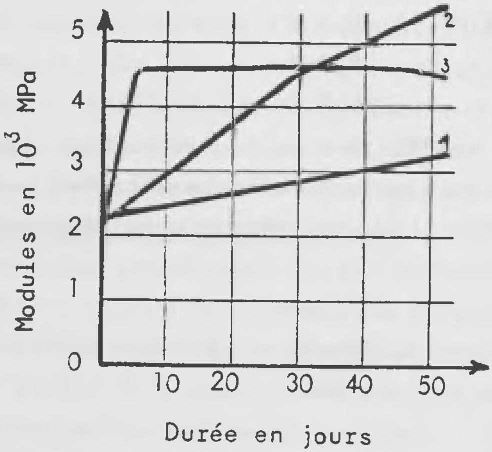
Cependant, cette composition de coulis est retenue pour le colmatage des gros vides de la maçonnerie.

VI.6.5 Durabilité des éprouvettes injectées :

Sur le gel de silice, obtenu à partir du silicate de soude, l'augmentation de la valeur du module d'élasticité dynamique longitudinal a été suivie, neuf éprouvettes prismatiques, 7 x 7 x 28 cm, en sable normal* ont été réalisées, puis injectées.

Trois modes de conservation après démoulage ont été étudiés :

1) en sable humide,
2) en salle sèche (50 % d'humidité relative - 20°C),
3) en étuve à 40°C



Les résultats sont donnés sur le graphique ci-contre. On note que le coulis maintenu à 40°C est constant après 7 jours à 4.400 MPa. Nous avons également examiné la durabilité de ces éprouvettes à l'essai de gélivité (NF B-10.513). On constate un comportement satisfaisant jusqu'à 50 cycles suffisant par exemple pour un emploi en rejaillissement. De plus, on observe une fragilisation des éprouvettes par dilution du silicate de soude non neutralisé.

VI.7 Application expérimentale in situ en l'Eglise de Sainte-Ménéhould :

VI.7.1 Injection du coulis de bentonite-ciment (chapitre VI.6.4) :

Pour l'injection de ce coulis, des trous de 30 mm de diamètre ont été forés sur 1 000 mm de profondeur avec une inclinaison de 45 °. (Le mur injecté mesure 1,10 m d'épaisseur).

La préparation du coulis est effectuée dans un agitateur à haute turbulence (photo ci-après).

* Sable servant aux essais mécaniques des ciments

Le malaxage du mélange est assuré par 4 hélices placées au fond de la cuve, et entraînées par un puissant moteur électrique. L'injection est faite par gravité, le coulis est versé dans un entonnoir débouchant dans les trous d'injection par l'intermédiaire d'un tuyau en caoutchouc.

VI.7.2 <u>Injection du coulis de silicate de soude (chapitre VI.6.4)</u> :

Des trous de 23 mm de diamètre et inclinés à 45 ° sur une profondeur de 700 mm ont été forés dans le pilier. Quatre injecteurs sont reliés à un distributeur à quatre voies, le tout relié à une petite pompe à main. (pression d'injection voisine de 0,15 MPa.).

VI.7.3 <u>Contrôle de l'injection</u> :

Préalablement aux travaux de forage des trous d'injection, il a été procédé à des mesures de vitesse du son en transparence. En général, les cavités existantes dans les murs et piliers n'ont pas permis la propagation des ondes sonores. Par contre, quelques jours après l'injection du coulis du gel de silice, nous avons pu mesurer des vitesses de propagation du son à travers la maçonnerie de l'ordre de 2.400 à 2.900 m/s.

Enfin, nous avons disposé sur les murs et piliers, des bases de mesures ainsi que des comparateurs, afin de suivre les déformations qui pourraient se produire pendant l'injection des coulis ; certaines de ces bases sont placées sur le parement en pierre, de part et d'autre, d'un joint de mortier, d'autres sont disposées de part et d'autre d'une fissure existante. Pratiquement aucune déformation notable n'a été enregistrée.

Le colmatage des murs de blocage a été également vérifié par des carottages jusqu'au coeurdes murs et l'on a constaté aussi une bonne diffusion des deux types de coulis.

Le coulis de bentonite n'obturant que les vides supérieurs au demi mm et ne diffusant pas dans le mortier de terre, et c'est le coulis de silice qui l'a imprégné.

VII. MISE EN APPLICATION DES PROPOSITIONS DU C.E.B.T.P.

VII.1 Partant de ces données des essais comparatifs furent effectués par M.J. NOEL et l'entreprise HORY qui avait pris part à titre d'observateurs à l'étude du laboratoire.

VII.2 M.J. NOEL a fait construire dans son atelier de Reims, rue des Capucins, 3 murets grandeur nature en moellons de pierre calcaire et craie maçonnés de mortier très maigre et terre mouillée. Après séchage, ces murets qui comportaient de nombreuses poches vides négligemment laissées en les montant furent injectés de coulis de béton bentonite.

Les résultats suivants furent observés :

après un mois de séchage un parement fut abattu et le blocage examiné, on peut constaté :

que le coulis avait bien pénétré les vides et interstices des murs même dans certaines fissures fines de moins de 1 m/m de section,

qu'il avait adhéré aux masses de mortier et aux blocs de blocage ainsi qu'à la face arrière du parement,

qu'il fallait les frapper fortement pour faire éclater les mottes qui remplissaient les vides et qu'à l'intérieur de celles-ci, la masse du coulis était fracturée en de nombreux morceaux par des fissures parfois larges de plusieurs millimètres comparables par leurs positions et leurs formes à des fissures de dessiccation.

L'entreprise HORY a travaillé in situ à l'église de Sainte-Ménéhould après constatation des résultats précédents. Le mur Ouest du croisillon Nord au-dessous de la fenêtre fut choisi pour l'essai à faire le mur était constitué de deux parements de gaize avec blocage intermédiaire, les coulis faits courant juin 80, après 3 semaines de séchage, 2 pierres de parement furent arrachées. Le coulis avait atteint leur face arrière, et le blocage examiné, celui-ci manifestait les mêmes caractéristiques que dans les essais précédents.

VIII. CONCLUSIONS
OPERATIONS A POURSUIVRE

Ces deux essais et leurs résultats positifs, que le procédé du coulis de ciment n'aurait pas permis d'espérer, indiquent les grandes lignes des opérations à poursuivre :

VIII.1 <u>Immédiatement</u> :

affinage de la formule du procédé par de nouveaux essais in situ diversifiés sur des natures de pierre différentes avec variation des dosages des composants en fonction des qualités de ces pierres, en même temps, chaque fois qu'il est possible de trouver un mur compatible, essai de la méthode de contrôle non destructive de la vitesse de propagation du son, tous les chantiers ne permettant pas une vérification "anatomique" d'ailleurs seulement valable pour la seule partie examinée. Il sera ainsi possible d'établir petit à petit, des tables d'utilisation en fonction des matériaux employés.

VIII.2 Dans un second temps :

améliorer le matériel de préparation des coulis et de contrôle : tonneau mélangeur ou mixeur plus facile d'emploi par les équipes de chantier.

Tuyauterie et entonnoirs en plastique à complèter par un dispositif d'insuflation d'air comprimé à employer dans des cas particuliers. (Mieux connaître la limite d'emploi pour la sécurité des murs).

Appareils de chantier pour la mesure de la viscosité et du retrait à perfectionner avec l'aide du CEBTP afin de permettre les vérifications et tests sur le tas, pour les mélanges : chaux + bentonite + sable ou autres,

essais en zone humide ou inondée, etc...

Recherches sur le traitement au silicate, pour le rendre opérationnel en toutes circonstances.

La première phase de cette recherche différée comporterait en priorité les traitements dans les zones sèches et saines en excluant pour le moment, sauf à titre expérimental dans des cas bien précis : les zones humides, les fondations,

les glacis et parties hautes des murs soumises à l'humidité,

le dégré de perméabilité du mélange bentonite ciment ou chaux étant à définir de façon plus précise.

Une confrontation des résultats pourrait être prévue dans un délai de deux ans.

VIII.3 Evaluation de ces ouvrages valeur série :

L'évaluation de ces ouvrages devrait être faite de façon la plus exacte possible dès maintenant afin d'éviter de détourner les entreprises d'un effort qui ne serait pas compensé équitablement, quitte dans deux ans à revoir aussi cette question.

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- Note sur les coulis de consolidation

  rédigée par Monsieur WALDSCHMIDT, Président de la Section scientifique et technique de la Compagnie des Architectes en Chef des Monuments Historiques.

ESSAIS DE LABORATOIRE SUR DES COULIS A BASE DE CIMENT

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M. FORTI *
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RESUME

La performance d'un coulis d'injection dépend de la dimension des vides à remplir et de quelques facteurs inhérants au coulis lui-même, comme la fluidité et la stabilité, qui sont déterminés, à leur tour, par la combinaison d'autres facteurs primaires, comme la quantité d'eau, la forme des grains, la présence d'adjuvants, la vitesse et la durée du malaxage etc.

La colonne d'injection développée au Laboratoire des Ponts et Chaussées paraît permettre une évaluation de l'injectabilité d'un coulis avec plus de précision que le cône de Marsh.

L'emploi de ciments à basse teneur en substances alcalines et de fillers peut améliorer les propriétés chimiques et mécaniques des coulis destinés aux maçonneries anciennes.

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Rome, octobre 1981

1. INTRODUCTION

Des injections sont appliquées assez souvent pour le traitement de sols instables ou la réparation de béton fissuré. Matériaux et techniques sont assez bien développés dans ces domaines-là. Grâce à ces expériences, de nombreuses injections ont déjà été exécutées dans des murs de monuments, mais pas toujours avec des résultats complètement satisfaisants, à cause des caractéristiques des matériaux mal adaptés à cet emploi. Les résines sont trop fortes et trop imperméables, les pâtes de ciment ont un retrait trop élevé et forment des substances alcalines qui peuvent provoquer des dégâts par cristallisation. En outre la pression nécessaire pour une bonne pénétration des matériaux n'est pas supportée par des structures faibles. Notre recherche s'est orientée vers des matériaux inorganiques injectables à basse pression, capables de s'adapter aux exigences de chaque cas spécifique.

Les caractéristiques importantes pour ces matériaux sont : une bonne pénétrabilité pour l'injection à basse pression, une période assez longue dans laquelle cette pénétrabilité reste bonne, un retrait de durcissement minimal, une bonne adhérence aux parois des creux à remplir, une résistance mécanique et une porosité comparables à celles du matériau à réparer, un coefficient de dilatation le plus possible proche de celui du matériau ancien, un apport dans les vielles structures d'une quantité minimale de sels solubles ou d'autres matières nocives. Le but principal de notre travail était d'étudier, par des essais préliminaires, la mise à point d'un système d'essais capables d'évaluer cette gamme de caractéristiques.

Le travail a été partiellement financé par des bourses d'étude allouées par la CEE à deux architectes de la "Scuola di Specializzazione per lo Studio ed il Restauro dei Monumenti" de l'Université de Rome, Faculté d'Architecture, et à un ingénieur civil de l'IStituto di Scienze delle Costruzioni de l'Université de Rome, où les essais mécaniques ont été exécutés.

La mesure de la porosité et l'analyse des substances alcalines ont été exécutées au laboratoire du C.N.R., Centro di Studio per le Cause di Deperimento ed i Metodi di Conservazione delle Opere d'Arte, Rome. Nous remercions Mme P. Rossi Doria e M. F. Guidobaldi pour leur précieuse collaboration.

2. L'ESSAI D'INJECTABILITE. DESCRIPTION

L'injectabilité est une caractéristique d'importance primaire, parce que si l'injection même n'est pas bonne, les autres caractéristiques n'ont plus aucune importance.

Aux Laboratoire des Ponts et Chaussées en France (Paillère, Rizoulières, 1978) on a développé un instrument (et essai) simple, qui permet d'avoir une idée comparative de l'injectabilité des résines synthétiquesn mais qui peut être utilisé aussi pour des coulis granulaires, comme une pâte de ciment. L'appareil est illustré dans la figure 1.

L'appareil a été légèrement modifié par C. Wheatley de l'ICCROM par l'introduction de deux barres d'acier et deux brides métalliques qui permettent de fermer la colonne du dehors. Dans la colonne originale les deux bouchons en plastique sont fixés dans la colonne de plastique elle-même. Avec cette modification les colonnes coûtent dix fois moins chers et on peut les sacrifier, sans trop de regret, pour l'exécution des essais mécaniques, qui impliquent le découpage en morceaux de la colonne injectée.

Nous avons appliqué cet essai ainsi : le coulis dans le récipient est injecté sous une pression constante de 0,75 bar dans un tube vertical en plexiglas (l = 35 cm, Ø = 2,5 cm), rempli de sable calibré (grains entre 1,00 et 1,70 mm), mouillé au préalable dans l'appareil même. La granulométrie du sable détermine la dimension des pores à injecter (environ 200μ dans ce cas-ci). Le ciment et les fillers des coulis sont passés dans un tamis avec maille 75μ.

Pendant l'essai, on mesure le temps et la hauteur de montée, la durée de l'injection et le volume de coulis sorti du sommet du tube. Les colonnes injectées sont conservées fermées pendant 28 jours, puis coupées en tronçons de 5 cm. Les cylindres obtenus sont extraits du tube et soumis à un essai de fendage (essai brésilien).

A l'aide des essais exécutés on peut déduire des modèles d'injection, c'est-à-dire isoler les différents paramètres déterminant l'injection et leur donner une évaluation relative. En même temps, les essais donnent de l'information sur les matériaux injectés, sur leurs qualités comme coulis.

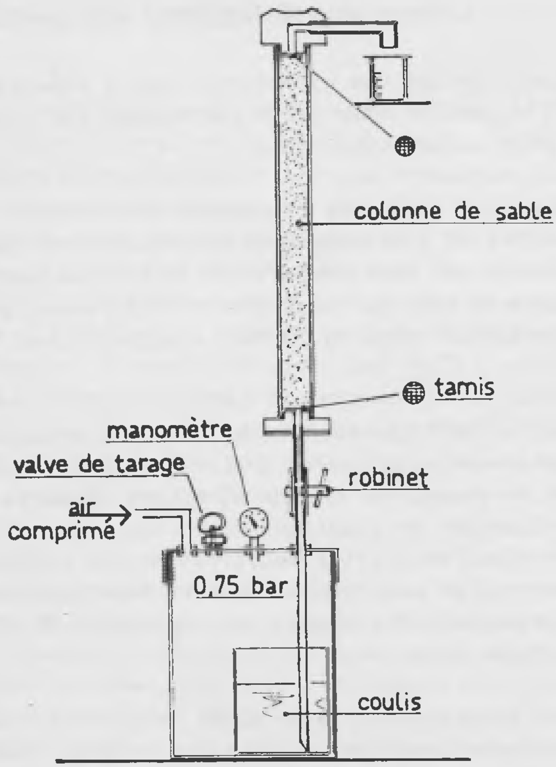


Fig. 1 Appareil pour la réalisation d'essais d'injection.

3. FACTEURS QUI DETERMINENT L'INJECTABILITE D'UN COULIS DE CIMENT

3.1 Contrainte d'écoulement

Dans le cas d'un coulis à base de ciment, ou d'une poudre insoluble en général, il ne s'agit pas d'un fluide parfait, c'est-à-dire newtonien, mais d'un fluide qui se rapproche du modèle de Bingham (Scott, 1975). Il faut dépasser une certaine contrainte de cisaillement pour commencer son écoulement; au dessus de ce seuil d'écoulement, la vitesse d'écoulement est proportionnelle au surcroît de la contrainte de cisaillement appliquée.

Cette contrainte d'écoulement est un facteur qui diminue la différence de potentiel déterminant l'injection dans une colonne de sable. Elle est proportionnelle à la surface de contact du coulis en mouvement avec son environnement fixe et donc aussi proportionnelle à la hauteur atteinte dans la colonne. La surface de contact étant très grande dans la colonne de sable, le seuil d'écoulement du coulis semble constituer le facteur le plus important dans la détermination de la hauteur de pénétration de l'injection.

3.2 Possibilité de blocage des pores de la colonne

Nous n'avons pas fait d'essais pour déterminer les critères de blocage des grains de ciment dans les pores du sable à cause de leur dimension. On peut trouver dans la litérature des valeurs de 1/10 pour le rapport entre les plus gros grains du coulis et les grains les plus petits du sable, ou de 1/3 pour le rapport entre les plus gros grains du coulis et les pores du sable. C'est la raison pour laquelle nous avons tamisé les ciments et les fillers avant usage. Nos essais ont indiqué aussi l'importance de la quantité relative de gros grains dans le coulis pour le blocage des pores.

3.3 Fluidité

Par fluidité on entend ici une généralisation du comportement mécanique compliqué du coulis. Les éléments de ce comportement (viscosité, seuil d'écoulement) sont déterminés en effet par les mêmes prémisses, mais pas toujours dans la même mesure. La fluidité est l'apparence externe de la friction interne dans le coulis. Le facteur le plus important déterminant la fluidité est évidemment la quantité d'eau par rapport au volume de solides. Mais il y a d'autres facteurs:

- la finesse de la poudre : la surface globale des grains d'une poudre plus fine nécessite une quantité d'eau plus grande pour l'englobement de tous les grains dans un film d'eau. Pour une fluidité comparable une poudre plus fine aura donc besoin de plus d'eau.
- la forme des grains : la friction entre des grains de forme très irrégulière sera beaucoup plus grande qu'entre des grains sphériques, par exemple. Pour une même fluidité ils doivent maintenir une distance plus grande entre eux, il y aura besoin de plus d'eau.
- la courbe granulométrique : une courbe granulométrique discontinue laisse des trous dans le tassement des grains, qui doivent être remplis d'eau. Cette eau n'a aucune influence sur la fluidité.
- l'hygroscopicité : des grains hygroscopiques absorbent de l'eau qui n'apporte rien à la fluidité.
- l'agglomération des grains : normalement les grains d'une poudre tendent à se conglomérer, à ne pas laisser passer de l'eau entre eux (à cause des forces d'attraction et de la tendance vers un état d'énergie minimale du système). Mais pour une bonne fluidité il faut que tous les grains soient dégagés et englobés par un film d'eau.

On peut obtenir une bonne distribution des grains par une agitation vigoureuse du mélange lors de sa préparation. Un autre moyen pour obtenir une meilleure distribution des grains est offert par des "fluidifiants", ou "réducteurs d'eau", qui sont des agents chimiques tensio-actifs qui diminuent la tension superficielle, sont adsorbés par le ciment (par les constituants calcaires) et adsorbent eux-mêmes des molécules d'eau (Rixom, 1978; Turriziani, 1972). Ils peuvent améliorer ainsi l'englobement des grains de ciment dans une couche d'eau, à condition qu'ils soient bien distribués et que les grains de ciment soient libres. On comprend facilement l'importance d'une agitation vigoureuse quand on emploie des tensio-actifs.

3.4 Stabilité

Dans un liquide hétérogène comme ces coulis à base de ciment, les composants plus lourds tendent à se séparer des composants plus légers: les grains se déposent sur le fond et l'eau vient à la surface jusque à ce que les grains soient tassés en équilibre. Il y a alors une quantité d'eau entre les pores du tassement et un surplus au dessus des grains. L'appareil d'injection prend le coulis du fond du récipient, là où il est le plus dense, où il est le moins fluide.

Ce sont la vitesse et la mesure de la ségrégation qui déterminent la stabilité du coulis. La mesure est presque uniquement fonction de la quantité d'eau en surplus du volume des pores. La vitesse de ségrégation est aussi déterminée par le surplus d'eau, mais il y a d'autres facteurs comme la forme des grains, l'affinité pour l'eau des molécules constituant les grains, et surtout la finesse de la poudre.

Avec une poudre fine les interstices entre les grains sont plus petits que dans une poudre à grains plus gros. En conséquence la vitesse de passage de l'eau entre les grains est plus petite et avec elle la vitesse de ségrégation. Le coulis à grains fins aura une meilleure stabilité que le coulis à gros grains. Une poudre à grains fins pourrait donc être utilisée comme stabilisateur d'un coulis (principe des "plastifiants" pour le béton). Une bonne distribution des grains dans le liquide favorise aussi la stabilité, probablement pour la même raison: les pores entre des conglomérats de grains sont beaucoup plus grands.

Equilibre fluidité - stabilité

Dans nos essais, nous avons constaté un jeu d'équilibre subtil entre la fluidité et la stabilité en vue de l'injectabilité. On peut présenter cet équilibre dans un schéma pour chaque coulis (fig. 2).

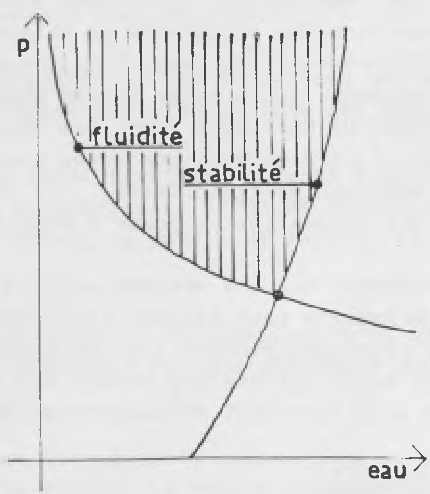


Fig. 2 Fluidité et stabilité d'un coulis d'injection.

Le schéma indique la pression à partir de laquelle la stabilité et la fluidité d'un coulis (en fonction de la quantité d'eau) permettent l'injection. La surface en dessus des deux courbes indique les possibilités d'injection du coulis. Le point d'intersection donne la pression minimale d'injectabilité et la quantité d'eau correspondante. Ce point se trouve dans les environs des conditions d'essais appliqués. Si le point se situe au dessus des 0,75 bar le mélange ne sera pas injectable; plus il est en dessous plus le coulis est facilement injectable. (Le schéma n'a qu'une valeur illustrative et ne correspond pas à des résultats d'essais).

4. PREPARATION DU COULIS ET CONTROLE DE LA FLUIDITE

4.1 Préparation du coulis

On a déjà indiqué l'importance de la façon de mélanger pour une bonne fluidité et stabilité. Nous avons constaté même la nécessité d'un malaxage à haute vitesse. Un premier mélange est fait à la main ou avec un malaxeur lent (comme pour la préparation d'un mortier). La confection du coulis même est faite par rotation à haute vitesse, pour bien séparer tous les grains et pour obtenir un mélange d'une excellente homogénéité. Nous avons utilisé un petit mixer de cuisine à capacité réduite, ce qui limitait nos possibilités. Nous avons constaté pourtant de grandes différences dans les résultants en changeant la durée de mélange violent, ou l'intervalle de temps laissé entre le premier mélange et le mélange violent, ou le temps laissé entre le passage dans le mixer et l'injection. On influence aussi les résultats en repassant le coulis une deuxième fois dans le mixer après quelque temps, on peut changer le moment d'addition du fluidifiant, ...: il y a un grand nombre de possibilités qui vaudraient la peine d'être explorées.

Il faut aussi indiquer ici une méthode pour la confection de pâtes de ciment stables à n'importe quel rapport Eau/Ciment: il s'agit d'un mélange fait pendant plusieurs minutes avec un mixer lourd, (jusqu'à 10000 t/min). Avec la haute vitesse les grains de ciment seraient activés de telle façon qu'ils restent en suspension dans l'eau.

4.2 Contrôle de la fluidité

Dans la pratique des injections de coulis à base de ciment la fluidité des mélanges est controlée par l'emploi du cône de Marsh. Il s'agit d'une espèce d'entonnoir qu'on remplit avec une quantité déterminé de coulis. On mesure le temps d'écoulement du liquide de l'entonnoir. On peut accorder au liquide une viscosité conventionnelle par la confrontation du résultat avec le temps d'écoulement d'un liquide newtonien de viscosité connue.

Dans nos essais nous avons constaté une anomalie entre les résultats avec le cône de Marsh et les résultats avec l'appareil d'injection. En effet, dans la colonne de sable le rapport de la surface des parois fixes sur la quantité de coulis passant est beaucoup plus grand que dans le cône. C'est ce rapport qui détermine l'influence du seuil d'écoulement sur l'écoulement. Dans la colonne le seuil d'écoulement est le facteur principal,dans le cône c'est la "viscosité" qui est la plus importante. Le cône de Marsh ne donne pas de valeur pour le seuil d'écoulement et n'est donc pas capable de substituer la colonne de sable pour le contrôle de la fluidité d'un coulis granulaire pour injection à basse pression. Quand la pression d'injection augmente, l'importance du seuil d'écoulement diminue. Le cône de Marsh reste un instrument valable dans le domaine des injections à haute pression.

5. RESULTATS DES ESSAIS D'INJECTION

Dans le laboratoire de l'ICCROM on a exécuté presque 90 essais d'injection de mélanges à base de ciment.

La plus grande partie de ces injections ont servi à mettre au point les facteurs règlant l'injectabilité des mélanges.

Vers la fin de cette série d'essais on a choisi deux types de ciment, un Portland pouzzolanique (325) et un ciment à haut teneur en silice (HTS),un filler, la poudre de marbre, et un fluidifiant,le gluconate de sodium.

Le gluconate de sodium a été préféré parce que les lignosulfonates ont donné des résultats peu satisfaisants dans les essais préliminaires.

Les ciments ont été injectés seuls, sans ou avec fluidifiant, et avec 20% et 40% de filler. Le ciment pouzzolanique ne peut être injecté sans fluidifiant. Egalement le ciment HTS avec 40% de filler n'a pas pu être injecté avec nos conditions de travail.

Toutes les colonnes ainsi traitées ont été découpées en 7 tronçons; les 6 tronçons inférieurs ont été soumis à l'essai brésilien de fendage.

Chaque coulis a été injecté 4 fois. Les résultats présentés dans la figure 4 sont les moyennes des 4 valeurs mesurées.

Dans la figure 4, le symbole $\overline{M}_{1-6}$ indique la moyenne de toutes les valeurs mesurées (tronçons de 1 à 6) pour chaque type de coulis.

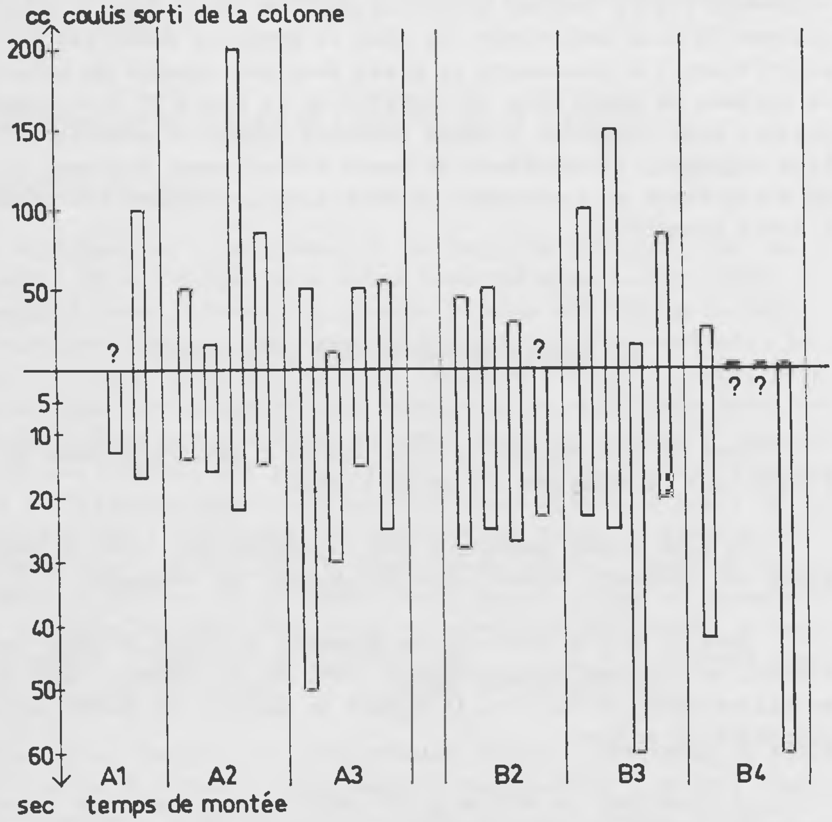
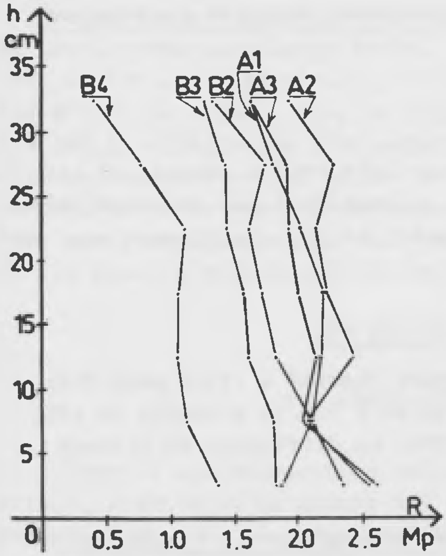


Fig. 3 Résultats des essais d'injectabilité



	A1	A2	A3	B2	B3	B4
7	1.62	1.92	1.62	1.32	1.26	0.39
6	1.79	2.26	1.90	1.74	1.43	0.76
5	2.00	2.13	1.92	1.62	1.44	1.10
4	2.18	2.20	1.99	1.72	1.57	1.05
3	2.16	2.42	2.13	1.82	1.61	1.05
2	2.10	2.08	2.03	2.09	1.81	1.13
1	2.33	2.54	2.60	1.87	1.83	1.37
$\overline{M}_{1-6}$	2.09	2.27	2.06	1.81	1.62	1.08

(valeurs en M·pascal)

Fig. 4 Résultats des essais brésiliens de fendage sur les colonnes injectés.

Composition des coulis (en grammes)

Symbole	Ciment	Marbre, Poudre	Eau	Na-Gluconate 30%	E/C
A_1	(HTS) 1000	-	590		59%
A_2	(HTS) 1000	-	500	3 ml	50%
A_3	(HTS) 800	200	480	3 ml	60%
B_2	(POUZZ.) 1000	-	550	3 ml	55%
B_3	(POUZZ.) 800	200	520	3 ml	65%
B_4	(POUZZ.) 600	400	490	2 ml	82%

Les résultats des essais d'injection sont discutés ci-dessous, divisés par matériaux.

Ciment Portland Normal 325.

Nous l'avons seulement employé pour initier les essais. Le sac était déjà assez vieux, les résultats ne sont donc pas représentatifs. Comme coulis eau+ciment on peut l'injecter le plus facilement avec un rapport E/C de 55%.

Ciment Portland à Haute Résistance : CPA 55 HTS

Ciment + eau (coulis A1) : Ce ciment (tamisé à 75μ) peut être injecté avec un rapport E/C de 59%. Tamisé à 53μ il a besoin de 63%, passé au tamis de 150μ il lui suffit 57%. La différence en finesse explique ces variations.

Ciment + eau + gluconate de soude (fluidifiant), (coulis A2): Le fluidifiant permet de réduire le rapport E/C à 50%, ce qui a sans doute un effet favorable sur le retrait. La stabilité du coulis ne change pas sensiblement. La résistance au fendage des cylindres durcis augmente de ca. 10%.

Ciment + eau + gluconate de soude + poudre de marbre (A3) : Le filler devrait réduire la résistance mécanique, rapprocher le module d'élasticité du coulis durci à celui de la pierre à traiter, et aura un effet sur le retrait, sur la porosité, sur la quantité de sels solubles.... Notre poudre de marbre est un peu plus grossière que le ciment. Un mélange de 80% de ciment et 20% de poudre peut être injecté avec 48% d'eau (sur le poids des solides). L'injection est presque aussi bonne que celle du coulis sans poudre de marbre. Avec un rapport ciment/poudre de marbre de 60/40 l'injection ne réussit plus, l'équilibre fluidité-stabilité n'étant plus favorable. Il est certain qu'avec une poudre de marbre ayant la granulométrie du ciment on réussirait facilement l'injection. La présence de 20% de poudre de marbre diminue la résistance moyenne au fendage de ca. 10%.

Ciment Portland Pouzzolanique 325

Ciment + eau (coulis B1) : Le coulis ne peut pas être injecté. Les meilleurs résultats sont obtenus avec un rapport E/C de 62%.

Ciment + eau + gluconate de soude (coulis B2) : Le fluidifiant déplace le point d'équilibre optimal entre fluidité et stabilité dans le bon sens. Le coulis a une bonne injectabilité pour un rapport E/C de 55%.

Ciment + eau + gluconate de soude + poudre de marbre (B3 et B4): Le coulis avec un mélange 80/20, ciment/poudre de marbre, 52% d'eau (B3) montre une légère meilleure capacité de pénétration que le coulis à 100% de ciment. Avec un rapport 60/40, 49% d'eau (B4) le coulis est à la limite de l'injectabilité, 50/50 n'y réussit plus. Encore une fois, avec une poudre de marbre de la granulométrie du ciment, toute la gamme de mélanges ciment-poudre de marbre devrait être aussi injectable. La résistance à la traction du coulis diminue de 10% pour le mélange 80/20, de 40% pour le mélange 60/40.

6. ESSAIS D'ADHESION

Dans le cas d'injection de coulis dans de la maçonnerie en brique, l'adhésion du ciment aux briques est évidemment un facteur essentiel pour la résistance mécanique de la maçonnerie entière.

Malheureusement, il est très difficile de mesurer l'adhésion par des essais de résistance au cisaillement de joints réalisés entre deux surfaces de brique. Les résultats sont souvent très différents.

Nous avons essayé une configuration très simple des échantillons, qui était déjà connue (Pierzchala, 1965). Il s'agit de trois blocs (4 x 4 x 16 cm.), coupés dans des briques commerciales de haute qualité, espacées l'une de l'autre de 2 mm. par de petits morceaux calibrés de laiton. Les coulis sont coulés dans les fissures à l'aide d'aiguilles d'injection.

Après la prise du coulis, une contrainte de cisaillement est appliquée par une compression sur le bloc central tandis que les blocs latéraux sont appuyés à des supports métalliques.

Une série préliminaire d'essais que nous avons effectué sur 13 échantillons avec des coulis à base de ciment pouzzolanique a donné des résultats assez décevants.

Deux échantillons se sont ouverts sous des contraintes occasionnelles, avant d'être soumis à compression; pour les autres on a obtenu des résultats de résistance au cisaillement entre 0,022 et 0,345 MPa avec des écarts considérables pour le même coulis.

7. POROSITE

Quatre tronçons centraux (les n. 4) coupés de quatre colonnes injectées (deux avec coulis de ciment HTS, avec ou sans filler,deux avec coulis de ciment pouzzolanique, avec ou sans filler) ont été soumis à la mesure de la distribution des diamètres des pores par le porosimètre à mercure.

Les mesures ont été exécutées par Mme P. Rossi Doria du C.N.R., en suivant la standardisation de cet essai approuvée par le comité italien NORMAL-F (Normal-F, 1980).

Les résultats complets sont reproduits dans la Table I. La valeur moyenne de deux mesures y est présentée.

Il paraît que le remplissage des espaces entre les grains de sable a assez bien réussi dans tous les cas (porosité totale au dessous de 20%, porosité plus large que 10 microns toujours au dessous de 15%). L'effet du filler sur la distribution des pores paraît négligeable.

Le coulis avec ciment HTS produit une porosité inférieure à celle déterminée par le ciment pouzzolanique, mais les distributions ne montrent pas de différences importantes entre les deux cas.

TABLE I <u>Distribution des diametres des pores</u>

Type de coulis	V% Pourcentage du volume total des pores (diamètre des pores en µm)											
	ρa (g/cm³)	P%	d < 0.05	d 0.05-0.1	d 0.1-0.2	d 0.2-0.4	d 0.4-0.6	d 0.6-0.8	d 0.8- 1	d 1 - 2	d 2 - 4	d 4 - 10	d > 10
A_2	2.12±0.02	18±1	32	17	13	8	4	1	1	4	2	4	14
A_3	2.13±0.02	13±1	39	23	6	5	2	1	0	1	4	5	14
B_2	2.11±0.02	19±1	43	24	7	4	2	1	0	2	1	3	13
B_3	2.07±0.02	20±1	41	26	7	4	2	1	2	6	5	2	4

<u>Note</u> Pour la composition des coulis voir la table attachée à la figure 4.
ρa = Poids specifique; P% = Porosité totale

8. SUBSTANCES ALCALINES EXTRACTIBLES

Sodium et Potassium ont été déterminés dans les mêmes échantillons sur lesquels ont été effectuées les mesures de porosité.

L'extraction a été faite par une méthode élaborée par S. Peroni et C. Tersigni (ICCROM, 1981). L'analyse des éléments alcalins a été exécutée par F. Guidobaldi du C.N.R. avec un spectromètre à absorption atomique.

Les résultats sont présentés dans la Table II. Chaque valeur est la moyenne de deux résultats.

La table montre de façon assez nette l'avantage de l'emploi d'un ciment à bas contenu en substances alcalines (le 55 HTS de Lafarge) qui permet de réduire à un tiers les sels alcalins que le coulis peut mettre en circulation dans les maçonneries anciennes, avec des effets souvent néfastes.

Le coulis avec ciment HTS peut quand même mettre encore en circulation plus de trois fois la quantité de substances alcalines extractibles d'un mortier de chaux pure (ICCROM, 1981).

TABLE II Matériel alcalin soluble dans les coulis

Type de coulis	Sodium (Na)meq/Kg	Potassium (K)meq/kg	Σ $Na^+ + K^+$ meq/kg
A_2	3.91	9.9	13.81
A_3	4.92	11	15.92
B_2	11.3	28.56	39.87
B_3	13.04	34	47.04

Note Pour la composition des coulis voir la table attachée à la figure 4.

ANNEXE - LISTE DES MATERIAUX EMPLOYES

- Ciment Portland artificiel (*)	CPA 55 HTS (haute teneur en silice) Ciment Lafarge, St. Cloud - France
- Ciment Portland (*)	Cemento 325 Italcementi, Colleferro, Italie
- Ciment pouzzolanique	Cemento Pozzolanico 325 S.A.C.C.I. - Amiterno, L'Aquila-Italie
- Poudre de marbre	Scarrocchio e Belardi 1 Via Capo d'Africa, Roma, Italie
- Carbonate de calcium	$CaCO_3$ polvere leggera 98,5% (contenuto di alcali solubili 1,12%) CODEX - Carlo Erba, Milano,Italie
- Gluconate de sodium	Gluconato di Sodio 98% Carlo Erba - Milano - Italie
- Sable	Sable pour préparation d'échantillons pour essais mécaniques. (Fractions utilisées 1.00-1,70 / 0,90-1,10). Satisfaisant aux normes Italiennes (D.M. 3.6.1968). S.I.S.A., Torre del Lago, Italie Une analyse de diffraction de rayons X (S.Z. Lewin - communication privée de données non publiées) a montré que les composants principaux de ce sable sont le -quartz et un plagioclase (probablement sanidine)
- Eau	l'eau de Rome a une dureté de 30°.

(*) Nous remercions les Sociétés Ciments Lafarge et Italcementi qui nous ont fourni gratuitement les ciments employés dans notre recherche.

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Section 3 :

Portland cement and special cements ;
their use in conservation.
Ciment Portland et ciments speciaux ;
leur utilisation dans la conservation.

STUDY OF HYDRATED CEMENT PASTES OF AGED CONCRETES FROM ACROPOLIS MONUMENTS

A.E.CHAROLA*

ABSTRACT

A study is presented of the hydrated cement pastes of two aged concrete samples taken from the Erechtheion on the Acropolis in Athens.

The samples are approximately 80-100 years old. They were analyzed by x-ray powder diffraction and infrared spectrophotometry, and their microstructure was studied by scanning electron microscopy.

The aged samples do not differ appreciably in composition or microstructure from that of more recently formed concrete.

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Introduction

One of the most widely used materials for construction in our age is Portland cement. There exists a vast literature on the subject dealing with its composition, structure and analysis.

The material is of a complex nature in itself, and manufacturing and setting conditions compound the number of variables that can affect its composition and microstructure.

Much work has been done analyzing the composition and structure of concrete in its first stages of solidification and crystallization (Brunauer, 1962; Walsh et al., 1974). Not so much has been done on aged concrete (Gebauer-Harnik, 1975).

Samples were obtained from the concrete used in the restoration work carried out by Balanos (1900-1933) on the Erechtheion on the Acropolis in Athens, and from the ca. 1900 concrete cast of a caryatid when the original was removed to the British Museum.

Experimental Procedures and Results

The samples were analyzed by x-ray powder diffraction and infrared spectrophotometry, and the microstructure was studied by scanning electron microscopy.

X-Ray powder diffraction of the Erechtheion sample showed the presence of: α-quartz and calcite as major constituents, and pseudowollastonite [α-$CaSiO_3$ (ASTM No. 19-248)], portlandite [$Ca(OH)_2$] and tobermorite [$Ca_5(OH)_2Si_6O_{16} \cdot 4\ H_2O$ (ASTM No. 19-1364)] or CSH-gel as minor components. Gypsum was also found in trace amounts. The main constituents, α-quartz and calcite, are probably the filler added to the concrete. In Figure 1, the fracture section of this concrete shows what appears to be a typical fracture of a limestone of homogenous grain size. Apparently marble powder was used to give a marble-like appearance to the concrete.

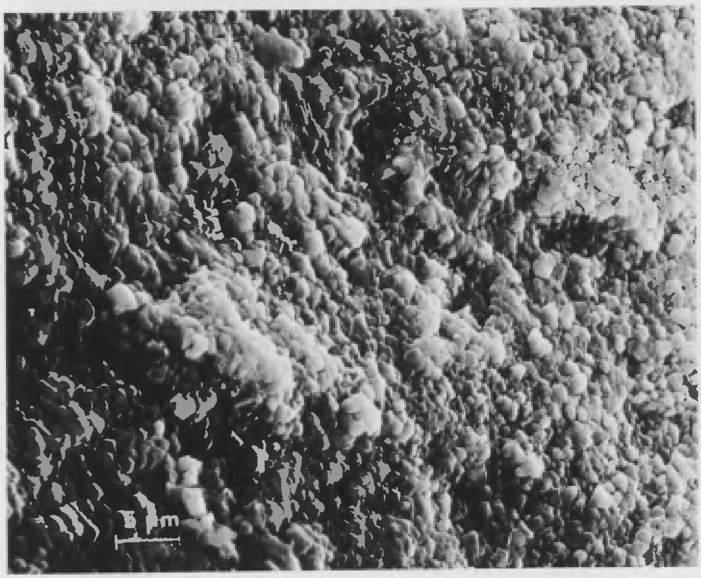

Figure 1 Scanning electron micrograph of the fracture surface of the concrete sample from the Erechtheion.

The sample was treated with 1 M HCl to remove the calcite. Figure 2 shows the appearance of a fracture section of the residual porous structure.

For x-ray diffraction purposes, the treated sample was separated into two fractions: the binder and the sand aggregate. The binder material was composed of a mixture of pseudowollastonite and tobermorite with some residual quartz. The aggregate proved to be quartz, as expected, also tobermorite and hillebrandite [$Ca_2SiO_4.H_2O$ (ASTM No. 9-51)] were also identified.

Figure 2 Scanning electron micrograph of the fracture surface of the Erechtheion concrete showing the porous structure left behind after treatment with 1M HCl.

X-Ray powder diffraction of the Caryatid sample showed the presence of calcite, portlandite and tobermorite.

The infrared spectra of KBr pressed pellets of both samples show the typical bands for calcite (υ_2 876 cm^{-1}, υ_3 1430 cm^{-1} and υ_4 712 cm^{-1}) (Farmer, 1974). The Erechtheion sample also showed the presence of quartz (Bensted-Varma, 1977), and in the Caryatid sample, because of the absence of quartz, the υ_{SiO_4} at 970 cm^{-1} from the calcium silicate hydrates was more obvious (Bensted-Varma, 1974). In this last sample, the presence of $Ca(OH)_2$ is evidenced by the very strong band at 3644 cm^{-1} (Bensted-Varma, 1974).

Discussion and Conclusions

According to some studies, the reaction of concrete with CO_2 will produce $CaCO_3$, SiO_2-gel and $Al(OH)_3$ (Sauman, 1971; 1972). This reaction is supposed to be complete for the silicate minerals encountered in hydrated cement pastes in about a month, its speed depending on the ambient conditions, but not quite so fast or complete for aluminates and silicoaluminates (these changing into a hydrogarnet phase upon liberation of $CaCO_3$). The microstructure of the samples should not however be altered appreciably, since the change is supposed to be a pseudomorphic one.

In our experience, and in the previously mentioned article on aged concrete (Gebauer-Harnik, 1975). the presence of silicates was identified. Not only were these silicates identified, but the presence of portlandite, $Ca(OH)_2$, was also established, and it would be expected that the reaction between CO_2 and $Ca(OH)_2$ would occur before any attack on the calcium silicates occurred. Thus, if some reaction occurred between the concrete and the ambient CO_2, this reaction was not complete even after 80 years of exposure to the atmosphere. Figure 3 shows a fracture through a book of $Ca(OH)_2$ platelets embedded in a calcium silicate hydrate matrix (Diamond, 1972). This micrograph is from the Caryatid sample.

The Erechtheion sample did not have such a high concentration of $Ca(OH)_2$ to start with, and furthermore, the presence of the $CaCO_3$ powder obscured the features of the binder, as can be seen in Figure 4.

Figure 3 Fracture surface of Caryatid concrete showing $Ca(OH)_2$ platelets embedded in CSH-matrix.

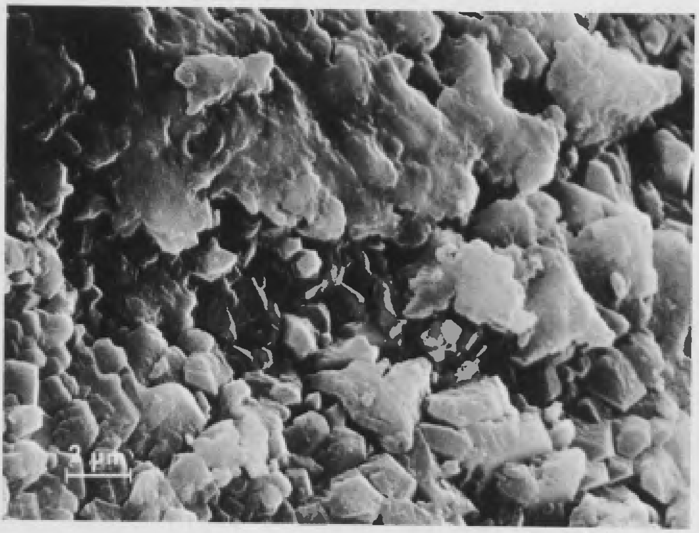

Figure 4 Fracture surface of Erechtheion concrete showing marble powder embedded in CSH-matrix.

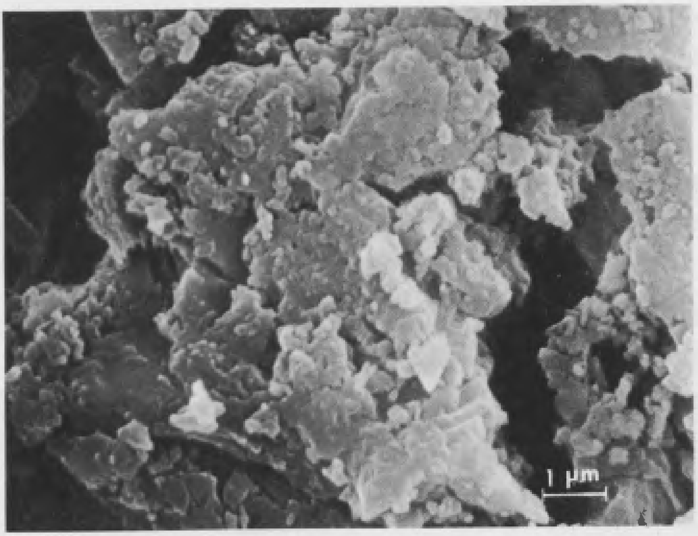

Figure 5 Fracture surface of Erechtheion concrete after treatment with 1M HCl showing the platy intergrowth of CSH.

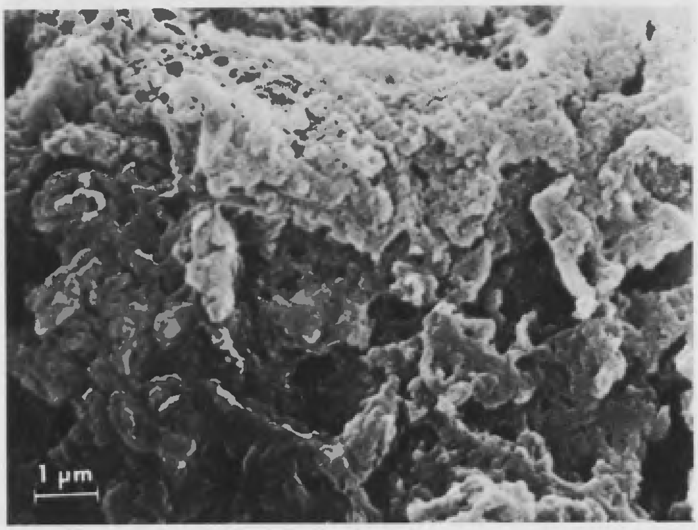

Figure 6 Fracture surface of Caryatid concrete after treatment with 1M HCl showing a similar microstructure.

When leached with HCl, the remaining structure shows a typical platy, pseudo-hexagonal appearance which may be attributed to the fibrous intergrowth of calcium silicate hydrates (Ciach et al., 1971). This feature can be seen in Figure 5, for the Erechtheion sample, and also in Figure 6, the corresponding micrograph for the Caryatid sample that was treated in similar manner.

In the Caryatid sample, as the microstructure was not obscured by the presence of marble powder, the fibrous growth of the calcium silicate hydrate forms can be seen, as shown in Figure 7.

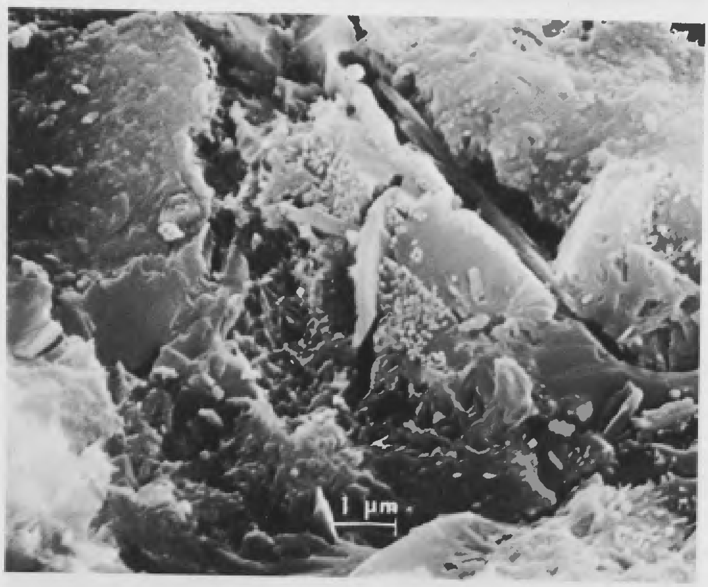

Figure 7 Fracture surface of the Caryatid concrete showing fibrous growth of the calcium silicate hydrates.

In conclusion, the study of two concrete samples, approximately 80 years old and aged under the same conditions, indicates: (a) that the carbonation reaction of concrete is not completed in that period of time; (b) that $Ca(OH)_2$ embedded in the concrete matrix can remain as such without reacting to form $CaCO_3$; (c) that no appreciable attack by atmospheric SO_2 is observed; (d) that the calcium silicate hydrates do not form well crystallized minerals even after such a length of time, as evidenced by the x-ray powder diffraction patterns and the IR spectra; and (e) that the microstructure of aged concrete is similar to that of more recently formed concrete.

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SOME TEACHINGS OF A ROMAN CONCRETE

FRANCO MASSAZZA * - MARIO PEZZUOLI *

SUMMARY

Analysis of concrete from the foundations of the Colosseum shows the intentional use of a low strength, low modulus, reduced shrinkage, good permeability material.

Factors affecting concrete performance in the restoration of ancient constructions are reviewed; mortars and concretes must show characteristics analogous or at least consistent with those of the ancient materials they are associated with.

It is necessary for the architect/restorer to specify the properties of modern mortars and concretes required to solve different restoration problems, just as the builders of the Colosseum did in the various parts of the building.

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SOME TEACHINGS OF A ROMAN CONCRETE

Franco Massazza and Mario Pezzuoli

1. INTRODUCTION

Romans largely used concretes especially in foundation works. The Roman concrete, as the modern one, was formed of more or less big stony elements bound by a hydraulic mortar consisting of a mix of lime and natural or artificial pozzolana.

As known, pozzolanic mortars harden owing to the lime-pozzolana water reaction. The formed compounds are the same as those obtained in today's portland, pozzolanic and blastfurnace cements, namely:
C-S-H gel,
$3CaO.(Al_2O_3,Fe_2O_3).Ca(SO_4\,/\bar{O}H\_7_2).12H_2O$ (solid solution formed by calcium monosulphoaluminate hydrate),
$2CaO.Al_2O_3.SiO_2.XH_2O$ (the so called gehlenite hydrate)(1)(2)(3)(4).

This fact must not surprise since all the usual binders essentially consist of CaO, SiO_2, Al_2O_3. Therefore, by reacting with water and for reasons of thermodynamic equilibrium, they must form the same compounds: calcium silicate and aluminate hydrates.

Of course this is only a simplification but at least, from the qualitative standpoint, it can be stated that ancient and modern hardened mortars are chemically similar. By contrast, physical and mechanical properties are different and especially mechanical strengths which are much lower than those of modern concretes.

Similarities and differences come out by examining a concrete sample cored from Colosseum's foundation and shown in figure 1.It consists of a grey, hard and compact aggregate having sizes up to 10 cm, surrounded by a grey, sometimes whitish, friable mortar. The latter contains sandy elements formed of the same grey coarse aggregate and redish burnt clay.

Figure 2 shows that the appearance of this concrete is not different from that of a modern concrete, apart from the aggregate size.

2. EXPERIMENTAL

2.1 Aggregate

A piece of aggregate was cleaned by brushing, ground and submitted to chemical and X-ray diffraction analyses. X-rays showed that this rock prevailingly consists of a feldspathoid, leucite ($KAlSi_2O_6$), associated with a monoclinic pyroxene, diopside ($CaMgSi_2O_6$), and a second feldspathoid, melilite $Ca_2(Mg, Fe, Al, Ca) (Si, Al)_2O_7$. This association of minerals is typical of an effusive rock belonging to the family of "leucitites" (5). A leucitite having a composition similar to the examined one is found in Rome, along Via Appia (near Cecilia Metella's tomb) in a place called "Capo di Bove", and is largely used for road stone pavements (6)(7).

Table 1 shows that the chemical composition of the two materials is very similar, therefore the aggregate in Colosseum's concrete is likely to come from the aforesaid zone.

2.2 Mortar

2.2.1 Structure and composition

Under the scanning electron microscope the mortar consists of irregular-shaped grains covered by very fine crystals forming a spongy structure likely composed of reaction products (see fig. 3). The mortar contains also lamellar crystals covered, in their turn, by the fine spongy new formations (see figures 4 and 5).

All the micrographs show a very little dense structure.

A portion of mortar was separated from the concrete, gently crumbled by hand and then sieved on a 40 μm sieve.

The chemical composition of the undersize is given in table 2.

The high loss on ignition, only partly attributable to the calcium carbonate content, shows that the mortar contains a high amount of hy -

drated material. The appreciable amount of calcium carbonate should be attributed to a partial carbonation of the lime used in the mix.

The X-ray diffraction analysis detected the following minerals:
- leucite, diopside, melilite, quartz,
- gehlenite hydrate and calcite.

Obviously the former come from the fine part of the aggregate and pozzolana, not retained on the 40μm sieve, and the two latter are the result of the pozzolanic reaction and the lime carbonation.

2.2.2 Porosity

Porosity was measured by a mercury porosimeter that also gave the pore radius distribution in the 75-750.000 Å range.

The mortar separated from the Roman concrete has a very different porosity from the one of an ordinary 28-day cured concrete. As table 3 shows, the former has a porosity about 4.5 times the latter and consequently a lower bulk density.

The graphs of figures 6 and 7 show the cumulative curves of the filled volume of pores and the percent distribution of the radii determined on the two mortars. It can be observed that the Colosseum's sample has a rather uniform distribution in the mesopore range (10^2 - 10^4 Å) whereas the control sample has a maximum at 350 Å.

2.2.3 Mechanical strength

The small size of available sample did not allow the Roman concrete to be submitted to loading tests. Nevertheless its mechanical strength could be evaluated by measuring the mortar hardness.

As known, the hardness of a material can be intended and defined in different ways not always in agreement and bound by precise relationships. However it is a function of the cohesion forces which bind the particles forming the material (8). In particular, the hardness tests based on the resistance opposed by the material against penetra tion are essentially miniature tests for tensile strength (9).

The hardness tests were performed on the mortar of Roman and control concretes by a durometer usually employed for measurements on metals. A 5 mm diameter sphere and a 31.2 kg load were used in the former case whereas a 2.5 mm sphere and 62.5 kg load were utilized in the latter. The obtained results were 3 and 60 kg/cm^2 Brinell hardness respectively.

Although these values cannot be easily transformed into mechanical strength values, they show that the binding mortar of the Roman concrete is much less resistant than the control concrete.

3. DISCUSSION

3.1 High porosity and low hardness show that the engineering properties of the examined concrete sample are lower than those of the concretes used in today's constructions.

Owing to its properties, the Colosseum's sample should be assimilated to the concretes usually called "gravel cements" and used in road construction. The latter have small cement contents (3-6% or 70-120 kg/m^3) and therefore have low mechanical strength (30-70 kg/cm^2), low modulus of elasticity, reduced shrinkage and expansion, good permeability (10).

The mortars used by the Romans, as well as the one which binds Colosseum's bricks, have generally higher hardness and therefore better mechanical properties. This leads to think that the particular characteristics of the concrete used in Colosseum's foundations were chosen and pre-established consciously on the basis of its intended use.

In fact foundations do not demand high strengths but rather moderate modulus of elasticity, reduced shrinkage and expansion, good permeability. This means that the Romans were perfectly able to suit the characteristics of mortars and concretes to any specific requirement.

This adaptability to varied conditions is typical of modern cements which consequently can be used to replace ancient mortars where, of course, it is necessary.

The essential condition that must be observed is that the properties of the new material must be as similar as possible to those of the material to be replaced or integrated.

The restorer is in the position of fully complying with this principle since he can make mortars and/or concretes of any required property, by selecting the materials and proportioning the mixes suitably.

Today's mortars and concretes are chemically consistent with the materials used in ancient buildings, since the chemical and mineralogical composition of the hydration products of the new binders is the same as the ancient ones. Therefore the restorer is only required to search for and to establish the physical and mechanical compatibilities between new and old materials by having resort to all the resources offered by modern technology.

The general rules which have to be followed to reach the best performances when cement is used in restoration are or should be known. Nevertheless, even if qualitatively, it is perhaps suitable to summarize the main conditions to be observed in order to obtain the desired properties.

3.2 The books on cement and concrete contain all the necessary indications to make mortars and concretes having the best performances and they must be consulted for the necessary information (11)(12)(13)(14)(15)(16).

They stress that the essential properties usually required from mortars and concretes are:

a) high workability
b) high mechanical strength
c) low shrinkage
d) good durability

a) Workability

The other conditions being unchanged, workability increases when:

- the w/c ratio increases
- the cement content increases
- roundish aggregates are used
- plasticizers are employed

As figure 8 shows (16), workability improves as the w/c ratio and the cement dosage increase since in both cases the total water content increases. However the two remedies have an opposite influence on mechanical strength.

Moreover workability can be considerably enhanced, without increasing the water content, by adding special organic admixtures to mortars and concretes, the so called plasticizers and superplasticizers. An example of their action can be seen in figure 9 (17).

b) Mechanical strength

The other conditions being unchanged, mechanical strength increases when:

- the cement dosage increases (within certain limits)
- the cement strength increases
- the w/c ratio decreases
- a suitable aggregate grading is used
- the particle shape of the aggregate is good
- compacting is accurate
- wet-curing is prolonged

Strength improves with increasing cement dosage (see figure 10) especially because homogeneity and compactness of concrete are enhanced, whereas it worsens as the w/c ratio increases (see figure 11) since at the same time the porosity of mortar or concrete rises.

The higher strength cements give better concretes as they produce pastes of higher cohesion force (see figure 11).

Moreover mechanical strength considerably depends on the grading curve and the shape coefficient of the aggregate. Grading must be as regular as possible so as to produce a mix that can be easily compacted to a maximum density. The shape of the aggregate particles must be as roundish as possible because it favours workability, mechanical strength and durability of concrete. The presence of elongated or flaky particles in excess must be avoided.

Obviously bad compaction and curing can considerably reduce the quality of mortars and concretes, even if they are proportioned and made with the utmost care.

c) Drying shrinkage

To reduce drying shrinkage it is necessary:

- to use a low w/c ratio, eventually by adding water-reducing admixtures
- to use aggregates having high modulus of elasticity
- to cautiously use products having expansive properties
- to wet - cure for long time
- to place the reinforcement (in concretes) correctly.

The drying shrinkage of mortars and concretes essentially depends on the amount and porosity of the cement paste and therefore it decreases as the cement dosage and the w/c ratio decrease(see figure 12)(18).

Moreover the unavoidable shrinkage of cement pastes can be reduced by a suitable choice of the aggregates and a prolonged wet-curing.

In many cases expansive admixtures can be a good solution to the problems caused by shrinkage. Nevertheless they must be used cautionsly.

d) Durability

To obtain the best durability it is necessary:

- to use a high cement dosage
- to use a low w/c ratio
- to add water - reducing admixtures
- to add air - entraining admixtures (to resist to frost)

- to use high chemical resistance - cements
- to carefully compact mortars and concretes.

To have a high durability, mortars and concretes must be as compact as possible. In order to reach this condition it is necessary to use high cement dosages, low w/c ratios, water-reducing admixtures and to carry out a good compaction. In special cases air-entraining admixtures (to resist to frost) or pozzolanic and blastfurnace cements (to resist to the chemical attack) need to be used.

These schemes show that different parameters act on certain requirements in an opposite manner:
for example the water content increase improves workability but worsens mechanical strength, shrinkage and durability. The cement content increase enhances durability and strengths, but also shrinkage.

As a consequence it is impossible to establish an only valid recipe for all cases and therefore the restorer is asked to suit the different parameters to the requirements of any work.

In any case, after hardening, mortars and concretes must show characteristics analogous to or at least consistent with those of the ancient materials which they are associated with. For this reason modulus of elasticity, coefficient of thermal expansion, mechanical strength, porosity and permeability must be carefully considered and checked.

Failures sometimes encountered in the cement use must be attributed to the non-observance of this compatibility, that is to a bad use of cement.

On the other hand, many experiences proved that today's cements can give an important contribution to the restoration work of the historical buildings.

It is only necessary for the restorer to be able to establish the properties of mortars and concretes, exactly as Colosseum's constructors did.

ACKNOWLEDGEMENTS

We thank Mr. Moccheggiani Carpano of the Superintendence of Monuments of Rome who kindly gave us the concrete sample cored from Colosseum's foundation.

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TABLE 1 - COMPOSITION OF THE AGGREGATE OF COLOSSEUM FOUNDATIONS AND OF "CAPO DI BOVE LEUCITITE"

Determination	Colosseum Aggregate	"Capo di Bove Leucitite"
L. o. I.	1.30	0.45
SiO_2	44.20	45.99
Al_2O_3	17.70	16.56
Fe_2O_3	9.30	10.15
CaO	12.16	10.47
MgO	4.43	5.30
SO_3	tr.	n.d.
Na_2O	2.36	2.18
K_2O	8.72	8.97
$CaCO_3$	2.50	n.d.
BaO	n.d.	0.25
P_2O_5	n.d.	0.56
TiO_2	n.d.	0.37

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TABLE 2 - COMPOSITION OF COLOSSEUM MORTAR - FRACTION BELOW 40 μm (FLORENTIN'S ATTACK)

L. o. I.	%	25.70
SiO_2	"	10.40
Al_2O_3	"	8.20
Fe_2O_3	"	1.00
CaO	"	22.90
MgO	"	0.90
SO_3	"	tr.
Na_2O	"	0.70
K_2O	"	1.30
n.d.	"	1.10
Florentin attack res.	"	27.80
$CaCO_3$	"	15.00

TABLE 3 - POROSITY AND DENSITY OF MORTARS SEPARATED FROM THE CONCRETES

SAMPLE	FROM COLOSSEUM	FROM MODERN CONCRETE
Porosity cm^3/g	0.2300	0.0494
Porosity %	36.05	10.77
Bulk density g/cm^3	1.543	2.180

FIGURE N° 1

Colosseum's concrete core

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FIGURE N° 2

Modern concrete

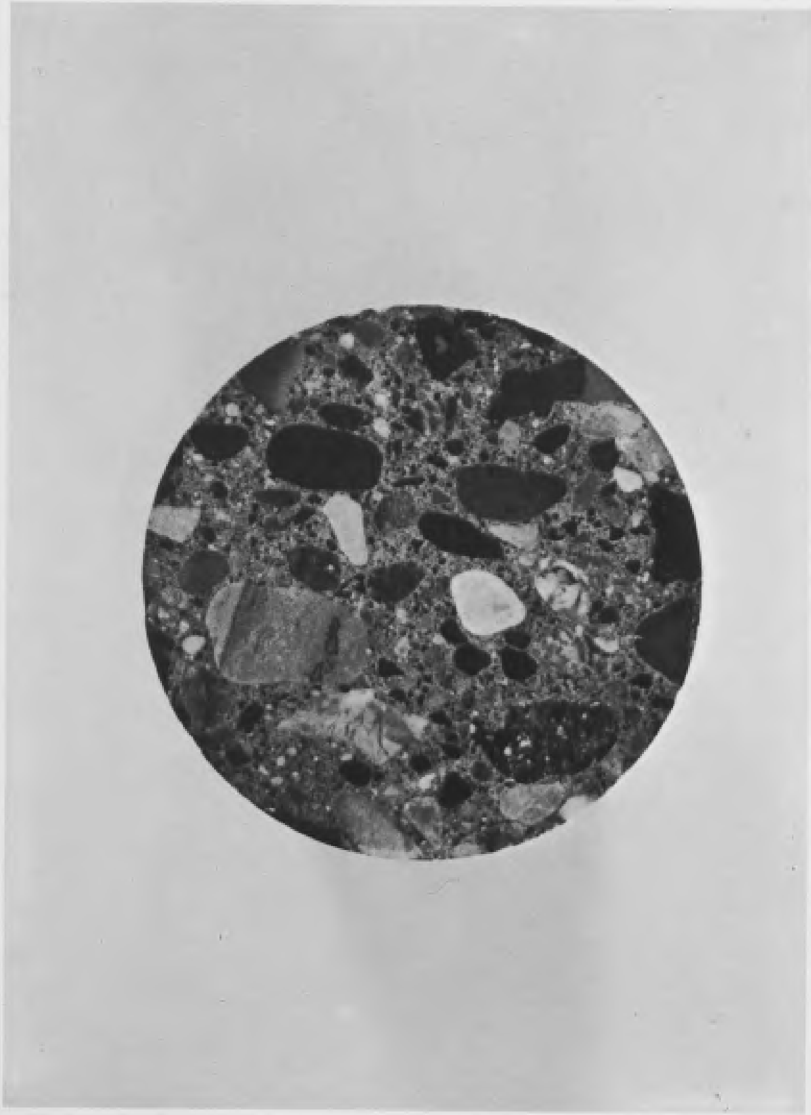

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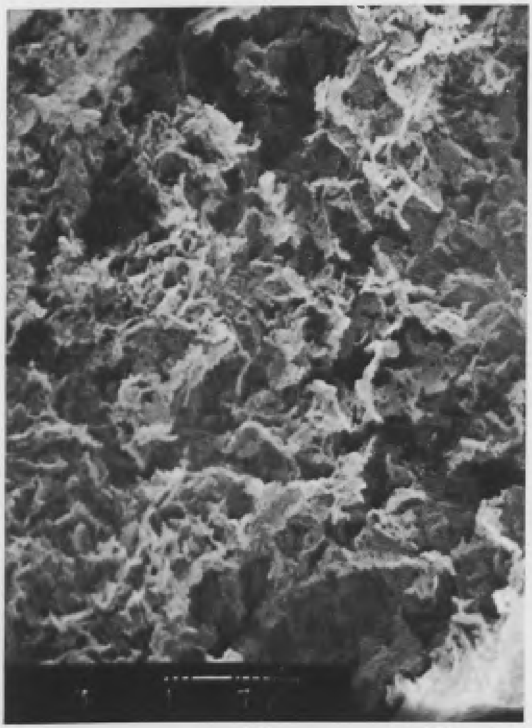
FIGURE N° 3

Colosseum's mortar SEI 10000 X



Spongy structure formed by hydration products

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FIGURE N° 4

Colosseum's mortar SEI 5000 X

Lamellar crystals covered by the spongy new formations

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FIGURE N° 5

Colosseum's mortar SEI 2000 X

Lamellar crystals covered by the spongy new formations

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FIG. 6: Mortar of Colosseum's concrete
Cumulative volume of pores Σ Vp and distribution of the pore radii % Vp

FIG. 7: Mortar of modern concrete
Cumulative volume of pores Σ Vp and distribution of the pore radii % Vp

FIG. 8: Influence of w/c ratio and cement content on concrete workability (16)

FIG. 9: Change in slump versus superplasticizer content with respect to cement

FIG.10: Influence of the cement dosage on the compressive strength of a concrete

FIG.11: Influence of cement strength and w/c ratio on concrete strength

FIG.12: Influence of cement content and w/c ratio on shrinkage (13)

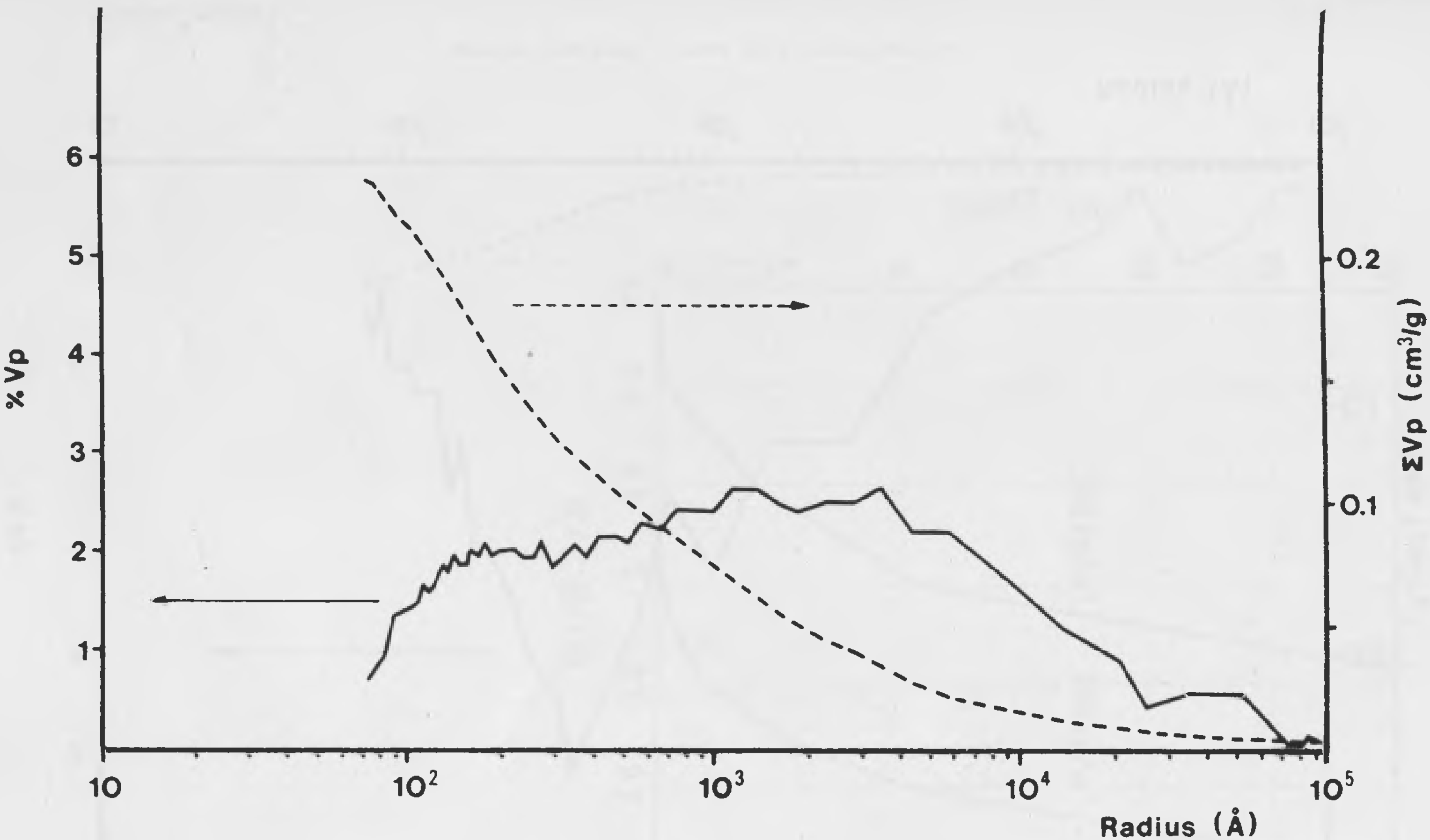

FIG. 6
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COLOSSEUM CONCRETE. PORE SIZE DISTRIBUTION

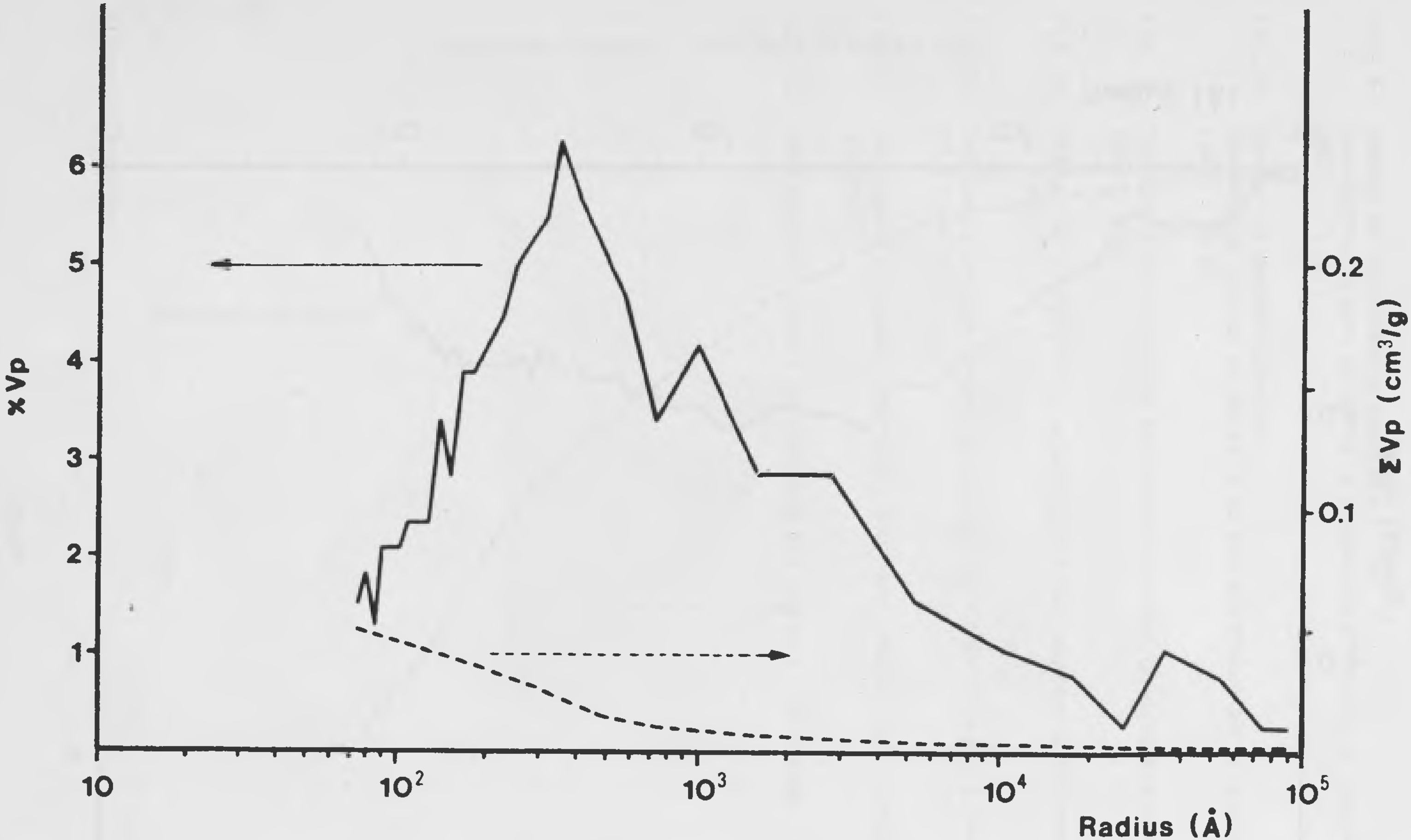

FIG. 7
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MODERN CONCRETE. PORE SIZE DISTRIBUTION

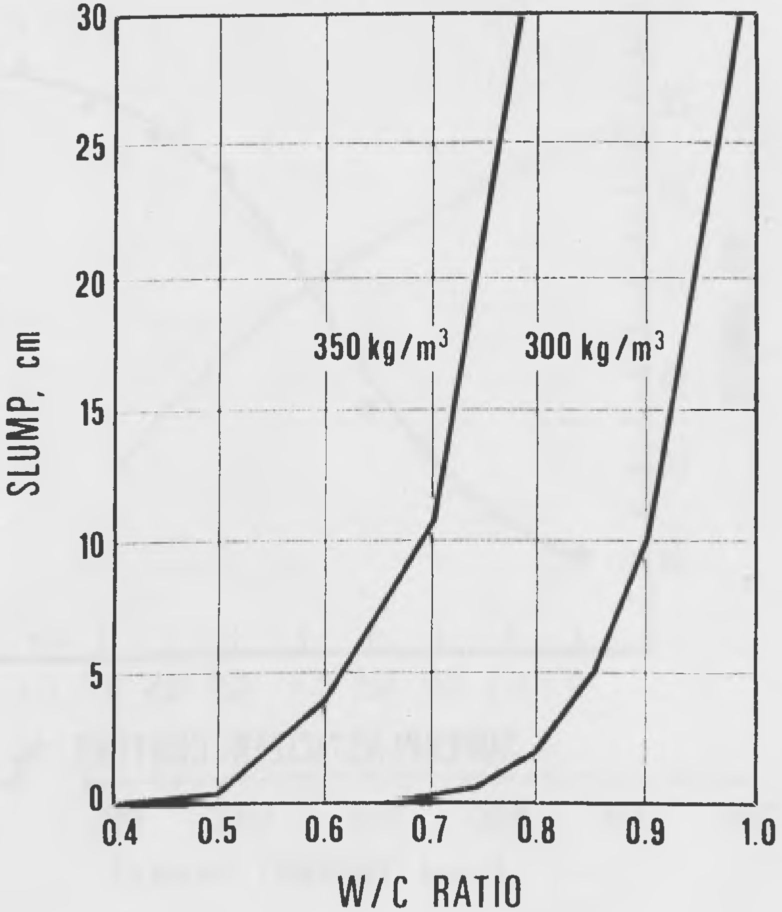

FIG. 8
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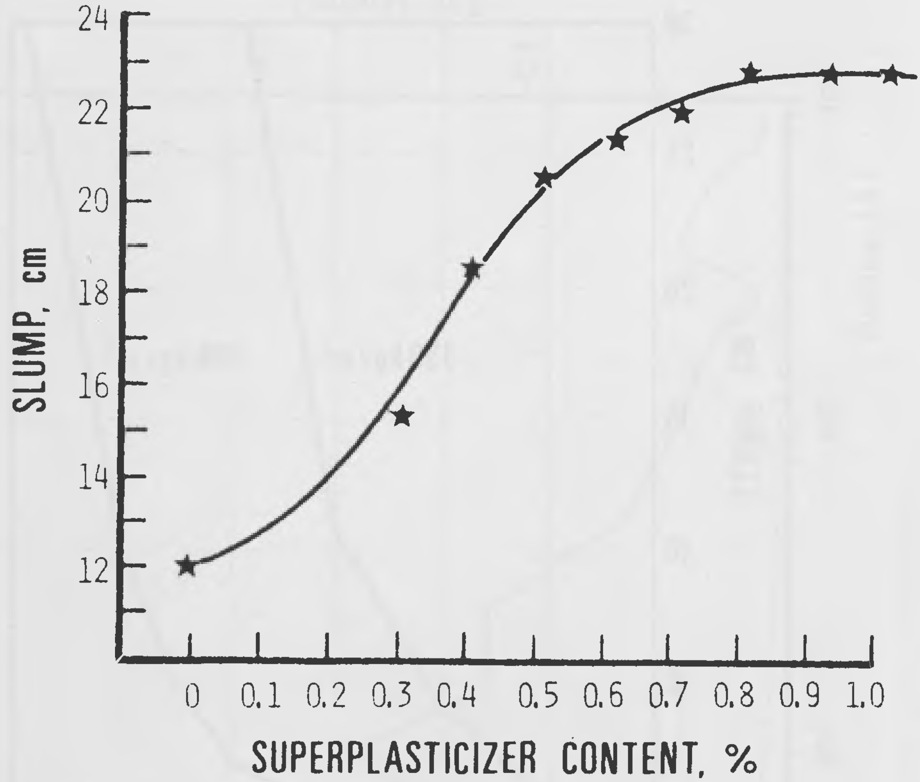

FIG. 9
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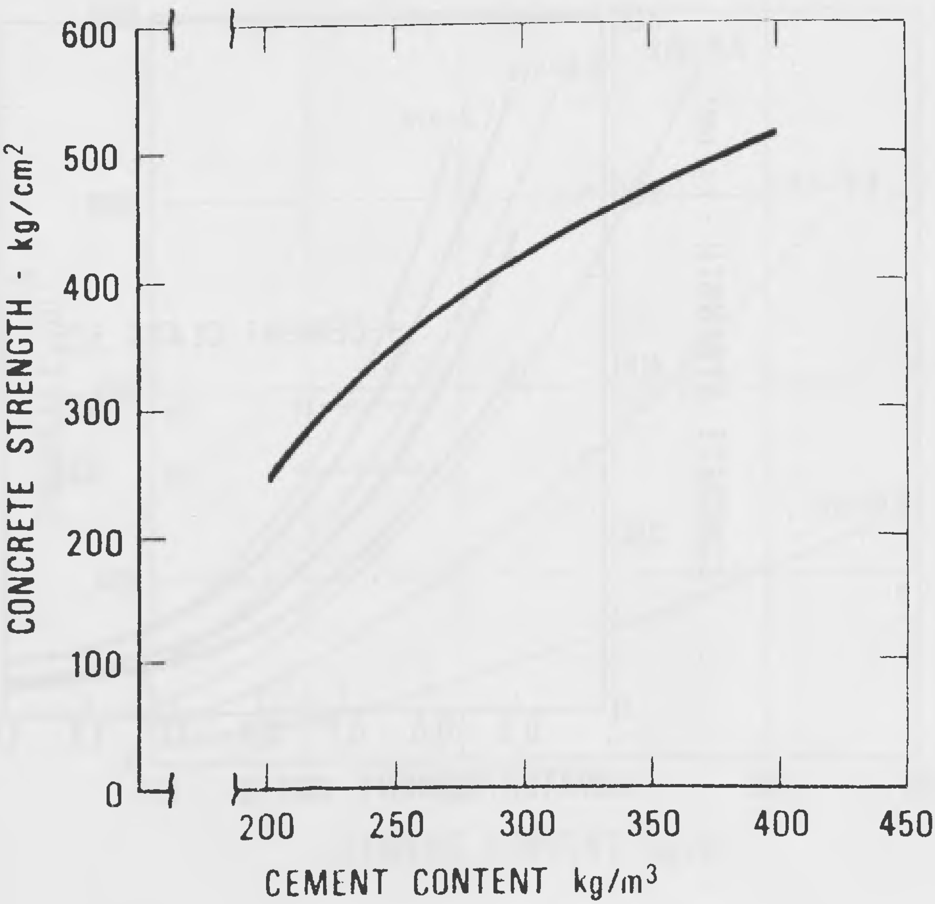

FIG. 10
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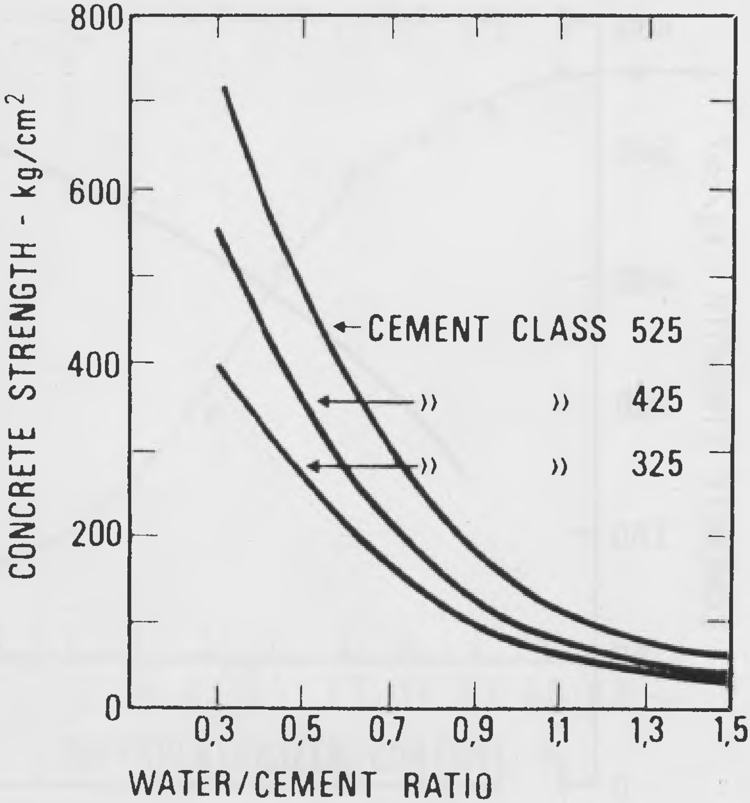

FIG. 11
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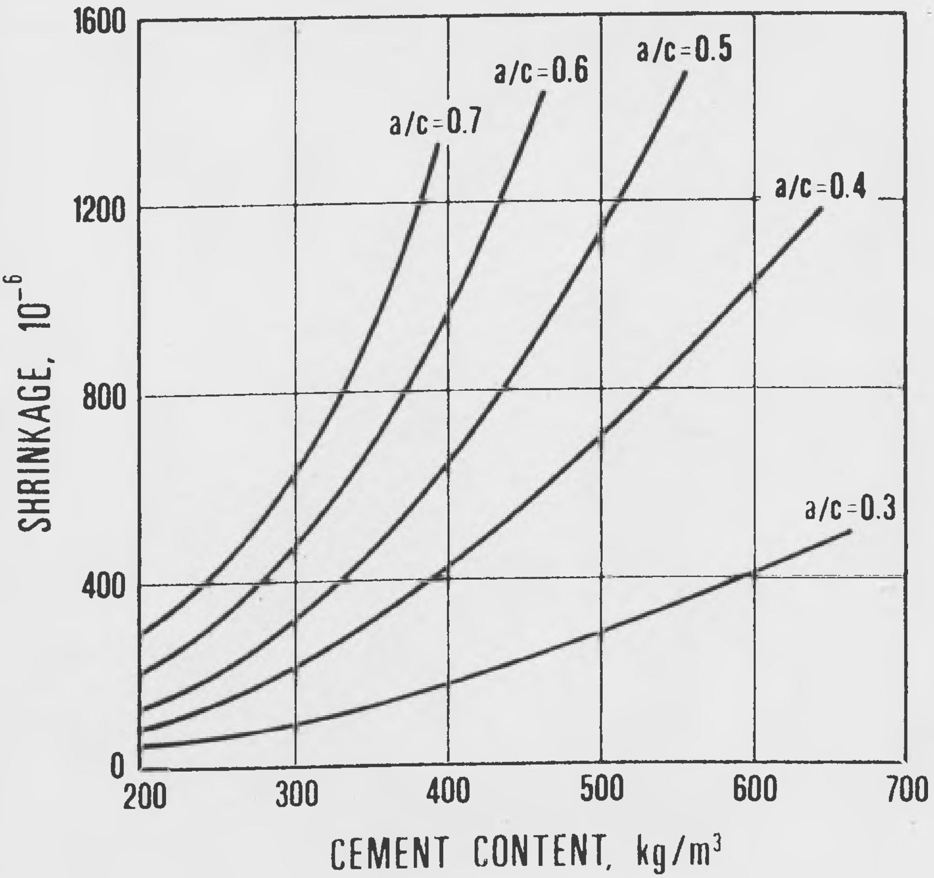

FIG. 12
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Section 4 :

Use of synthetic resins in mortars and grouts.
Utilisation des résines synthétiques dans les mortiers et les coulis.

THARROS (ORISTANO, SARDINIA): PRESERVATION OF THE PUNIC DITCH; METHODOLOGICAL CONSIDERATIONS.

Pio BALDI (^)

SUMMARY

Methodological hypothesis of this restoration is that it is necessary to neutralize the causes of damages on the monuments rather than repair their effects. It is proposed, therefore, to program the conservation circumscribing the factors wich cause deterioration and proceeding with little maintenance operations within short time intervals.

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Août 1981

The remains of the Punic fortifications at Tharros make up an imposing complex situated on the little peninsula of S. Giovanni di Sinis in the sorrounding of Oristano, Sardinia. Recent studies (1) have shown that Tharros was between the eigth and the seventh centuries BC a wealthy commercial centre and a trade call of the Phenicians; later, under the carthaginian dominion (since 500 BC approximately), it acted as a very important military base.

The most relevant feature of the said fortifications is a defensive ditch, about 100 metres long, bounded by thick walls of polygonal basalt blocks carsely squared. The preservative intervention we are going to describe here was effected on a portion of the above mentioned walls. Before entering into detail about the techniques employed, which is the subject of the present Symposium, we shall expound some methodologic hypotheses upon which the intervention was planned. It must be stressed that the relevance of such hypotheses is mostly theoretical and that consequently their applicability in this and in other restoring operations is naturally conditioned by the physical environment.

As to the method, therefore, every preserving operation is obviously aimed at the primary purpose of retarding the decay, caused to the object to be preserved by numberless external factors.

Every object is to be viceversa considered as in the condition of perennial lack of balance determined by the thermodynamic laws, so that its "useful life" consists of a measurable (and therefore non-infinite) period of time; its duration is a function of two variables which may be controlled to a certain extent: the attack by the external factors of deterioration and the effectiveness of the preserving interventions.

Since we are talking about cultural objects (that is about resources which cannot be reconstituted), we may add that the acceptable duration of their useful life is extremely short and tends to coincide with the time interval during which the object will not lose its distinctive features, corresponding to historico-esthetical characters. We could likewise assert that the maximum admissible level of degradation for a cultural object is extremely low.

It may be useful to visualize these rather obvious statements by means of the analytic reference by a Cartesian plane (diagram 1). Let us put time (t) on the abscissae axis and a quantity d, which we shall call deterioration level, on the ordinates axis. Such a quantity is the sum of the several external factors constituting the causes of degradation: d_1, d_2, d_3, ... d_n.

Some of the factors are of a meteoclimatic origin; rain, wind, frost, temperature range; some derive from chemical attacks of different nature (atmospheric pollution); some may be induced by physical phenomena: humidity, natural or artificial electro-magnetic radiations, movements of the ground; some may derive from biological agents or even from the direct action of man; etc.

The trend of time increase of degradation is schematically shown in Diagram 1. It should be pointed out that diagrams like this have been already published elsewhere in connection with studies on the programmed maintenance of buildings and on the concept of reliability, durableness and total cost of building works; it should also be pointed out that in other branches of industry the concept of programmed maintenance is by now acquired (2). But since our case, as aforesaid, is about cultural goods and not about simple consumer goods, we have to deal with different considerations: in the first place because acting on the causes of degradation seems to be more important than obviating to its damages (i.e. its effects), in order that such effects may be confined through suitable preventing actions; in the second place because the primary necessity of the preservation of the historical dosument mav. within limits. be detrimental to the effects of the total intervention costs and therefore the maintenance cannot be programmed to the only purpose of optimizing the general costs in relation to the capital invested.

Let us come back to our diagram. The degradation curve, which at the beginning tends to differ slightly from the horizontal, takes then a parabolic shape due to the acceleration and the self-excitation with time of the phenomena.

The ordinate d_{max} corresponds to the degradation level beyond which the object loses its physical characteristics. The abscissa t_{max} corresponds to the useful life of the object, but in the case of cultural properties the "acceptable duration" is likely to be obviously shorter.

The interventions of preservative restoration on the object result in a broken saw-toothed curve as in Diagram 2. Segments t_a, t_b, t_c, are intervals of time between the different interventions: for each of them the degradation level abates, i.e. the state of conservation betters. Anyways the intervention does not generally eliminate the whole of the accumulated degradation, thus $d_a < d_b < d_c$ etc.

Generally speaking the "useful life" of an object will increase as tendency of the degradation curve to pitch decreases, that is as much as we shall be able to control and limit the aggressive action of the external factors of decay (Diagram 3). In other words, in the presence of aggressive and uncontrollable deterioration factors, and therefore with the degradation curve tending to pitch rapidly, the duration of the useful life may be increased through rather strong interventions of preserving restoration and its curve will be of the kind described in Diagram 4.

The fact that we are referring to operations on cultural properties and not on simple consumer goods brings forth one more consideration.

Every intervention of preserving restoration, even the most respectful of the existing conditions, may result to be traumatic and there is a risk that some fragment or some documental episode may be lost. Particularly, the risk involved by a restoring operation is proportional to its radicalness. In other words, the deeper does ordinate d (degradation level) abate, the greater will be the risk of "empoverishing" the cultural object.

It derives that (Diagram 5) the Δd corresponding to the difference between d_a (degradation preceding intervention) and d_b (degradation following intervention) must be as small as possible.

It also derives that in order to prolong the "useful life", i.e. the preservation of the cultural object, we must put in the first place the control and limitation from the outside of the causes of degradation, that is of the aggressiveness of the external deteriorating factors. If we accept such premises, the hypothetical optimal trend is the one represented in Diagram 5, which conciliates the exigency to protract the duration of the cultural object with the necessity to avoid altering its nature.

This is the same as saying that a correct and effective preserving action must put in the first place a series of "soft" interventions, even at short time intervals; but they should be more similar to maintenance operations than to restoring ones. In the same time we must turn our efforts towards the identification and the control of elements d_1, d_2, d_3, ... d_n, which represent the progressive degradation factors.

Our aim was the careful identification of soft preventive interventions which ought to be integrated by periodical maintenance operations; we must avow that this was only partially possible, given the physical situation of the wall where we had to operate; but this will be the subject of the following contributions.

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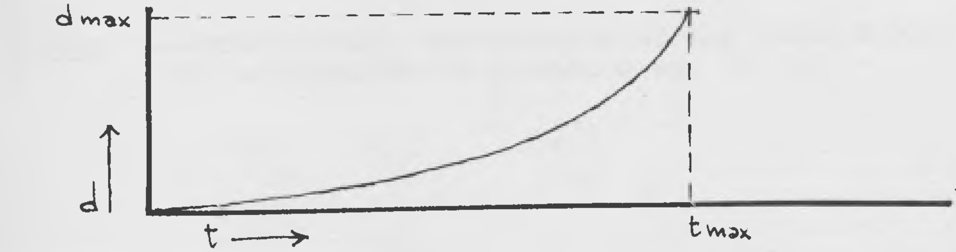

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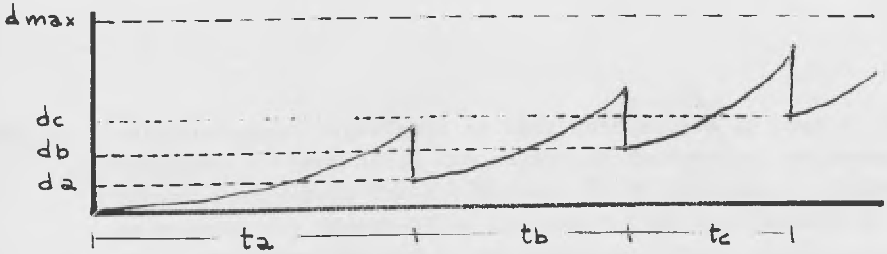

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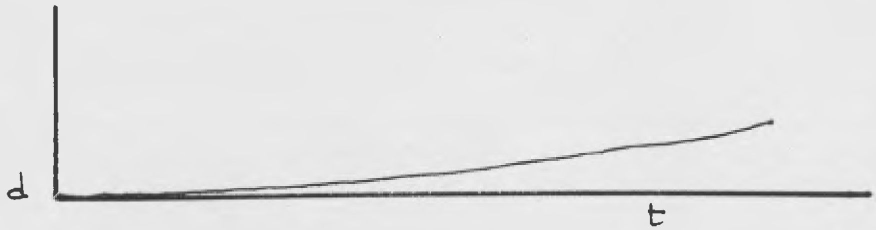

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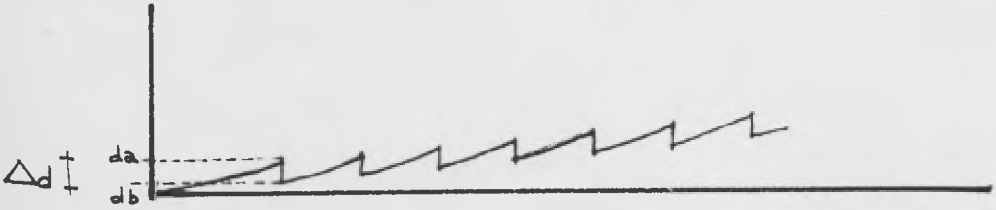

5

TITLE: THARROS (OR) : CONSERVATION OF THE PUNIC DITCH. DIVERSIFICATION OF TECHNOLOGIES.

AUTHOR: MARIO LOLLI-GHETTI

SUMMARY: Methodological hypothesis of this restoration is that it is necessary to neutralize the causes of damages on the monuments rather than repair their effects. It is proposed, therefore, to program the conservation circumscribing the factors which cause deterioration and proceeding with little maintenance operations within short time intervals.

FOOTNOTE: ARCHITECT TO THE ARCHAEOLOGICAL SUPERINTENDENCY OF LAZIO.

TECHNICAL REPORT

The work of consolidation and restoration here described is aimed at a first sector of the third defensive line of the northern fortifications to the Phoenicio-Punic town of Tharros in the province of Oristano. This defensive line, which has only been excavated in part, consists of a rampart, ditch and inner curtain wall.

The ditch, explored for a length of 70.00 m., has an average width, at its mouth, of 10.00 m., with a tendency to grow narrower towards its base. During the Roman imperial period it was occupied by a necropolis, utilizing a thick stratum of infill consisting of heterogeneous material, in large part derived from the adjacent walls.

The rampart delimiting the ditch on the downhill side had its inner side contained by a counterscarp wall built out of large polygonal blocks of black basalt; this was excavated to a maximum height of 2.70 m., though at least a further metre still underground should be added to this.

The curtain wall delimiting the ditch on the uphill side is preserved to a maximum height of 6.00 m., with an average width of 4.00 m. It consists of a thick rubble core of roughly shaped stones embedded in mud and contained between two revetments built out of polygonal basalt blocks, of which the outer one resembles the counterscarp wall.

Examining the area comprised by the intervention in greater detail, with particular regard to its existing state of conservation, the situation encountered is as follows:

Counterscarp wall

In the sector under review, the counterscarp wall delimiting the ditch on the downhill side preserves much of its inner facing in polygonal blocks of local basalt. This wall, which here rises from the present downtrodden level of the ditch to a height of approximately 2.00 m., is markedly out-of-plumb, and is currently being shored up by provisional wooden supports.

This out-of-plumb was presumably caused by the pressure of the earth comprising the rampart behind, not offset by the unstable consistency of the revetment and the inner rubble core, its stone components simply embedded in earth without any binding materials, which were not called for by the ancient constructional system.

In fact, the weight of the overlying materials was in itself sufficient to ensure the structure's equilibrium. But with the gradual disintegration of the wall complex as a whole in the course of the centuries, the overall system of static equilibria was disturbed, with the consequences described above.

Inner curtain wall

The condition of the curtain wall to the rear of the ditch differs from that of the counterscarp wall. Approximately 4.00 m. wide and 3.00 m. high, it consists of an inner core composed of roughly squared stones and two outer revetments in basalt and sandstone, whose existing state of conservation does not give rise to any immediate concern.

The wall is, however, being subjected to pressure by a deposit of detritus heaped against it which, due to its weight and lack of cohesion, could cause subsidences in its structure in the future.

The top of the wall, like that of the counterscarp wall, is broken, fragmentary and discontinuous, and thus offers easy penetration to the most damaging climatic and meteorological conditions.

Ditch

The stretch of ditch delimited by the walls described above, with a surface width of approximately 8.00 m., has only been excavated down to the settlement level of the Roman necropolis. To the latter belong a funerary monument built against the inner curtain wall and a number of small coffin-shaped tombs of extremely rare and distinctive type, built out of brick, rubble and plastered stonework.

Disintegrated walls in the eastern sector

It should be added that, outside the area considered of priority concern due to its disquieting lack of static equilibrium, a situation of grave deterioration was ascertained in extensive portions of the walls delimiting the ditch in the eastern sector. Devoid of their original revetment in polygonal basalt blocks, which have been almost totally removed or have collapsed into the ditch, these stretches of wall now consist in fact of their exposed inner core, comprised by assorted loose rubble of varying size embedded in earth and completely devoid of binding materials.

Investigations confirmed that this shapeless mass of wall is being subjected by climatic and meteorological agents to a perceptibly accelerating process of erosion and disintegration.

METHOD OF INTERVENTION

In response to the state of the structures described above, it was felt essential to adopt, as a priority commitment, an approach which would, in its techniques, materials and structural models, respect the existing situation to the utmost degree. This meant avoiding the introduction, within a static and constructional system currently in an unstable condition, of elements which, either due to their physical properties in relation to the materials used in antiquity or the dynamic of the static forces they could trigger off, might clash with and cause disturbance to the ancient fabric, with the consequence of provoking a series of constraints which would, in the long term, be both damaging and difficult to control.

On the basis of these considerations, the option of intervening with the methods conventionally utilized for consolidation was rejected, since such methods could cause far-reaching and irreversible alterations to the antique structure.

The utilization of cement was therefore ruled out, both in the form of liquid injection with a view to reconstituting the cohesion of crumbling masonry, and in the form of a continuous resistant shield with a view to containing the pressures exerted on it. This was prompted by several reasons: firstly, the tendency of cement to transfer soluble salts to the structures in contact with it; secondly, the excessive difference in its degree of permeability; and thirdly, the danger inherent in the introduction of a highly resistant material in a weaker structure, which would bear the brunt of the subsequent stresses induced, thus accelerating its process of decay. It is easily understandable, moreover, how a massive use of cement could seriously obstruct subsequent archaeological investigations or technical adjustments to the conservation programme.

For similar reasons, the option of consolidating disintegrated stonework by means of its absorption with epoxide resins was also discarded, since the use of such materials would not only fail, in this case, to guarantee effective results, but would clearly contrast with the building techniques originally used; in fact, as has already been pointed out, no evidence of the utilization of mortar or binding materials has been found in the walls present in the area of intervention. Furthermore, the application both of cement and resins in the form of widespread injection presupposes thorough preliminary cleaning either with water or pressurized air: a process which would inevitably cause serious damage to the structure in question.

Continuing our examination of the most appropriate conservational methods to be adopted, it has been thought advisable to seek a different protective finish to the top of the walls as an alternative to the rounded concrete capping normally utilized for archaeological restoration. Capping of this type constitutes in fact, in the majority of cases, an extraneous element[4] which forcibly completes the fragmentary structures to which it is applied through disturbing geometrical configurations.

On the other hand, there is no doubt that it does represent the least damaging solution hitherto devised for protecting the tops of walls from infiltration by water and the disintegrating action of atmospheric agents, in view of the fact that even the surface application of binding materials such as resin has so far failed to produce satisfactory results.

In the case in question, however, the option of capping of this type is automatically excluded both on practical and aesthetic grounds. In the first place, it is inconceivable to posit the casting of the mortar comprising such capping on a base of loose earth and fragmented stonework, since it is clear that a protective construction of this type would lose its effectiveness in a very short time. In the second place, in order to protect the top of the inner curtain wall with this system, it would be necessary to cast concrete caps from four to five metres wide. This, besides assuming an aesthetically unacceptable and incongruous aspect, would prevent a visual interpretation of the wall by concealing the various phases in its construction, which are clearly identifiable at the present time.

The feasibility of reconstructing, even in part, the polygonal revetment wherever it has collapsed by replacing the blocks which have fallen into the ditch, was also excluded, for two reasons. Firstly, due to the self-evident impossibility of exactly determining their original collocation (thus working by guesswork). And secondly, so as to avoid ambiguities in the structure's interpretation by restoring it with ancient material (and thus confounding the original with the restored fabric).

On the other hand, the option - though theoretically viable - of substituting the containment effect of the original revetment by a structural intervention carried out with visibly modern techniques and materials (reinforced concrete wall, brick curtain, block facing, etc.) would constitute an "authoritarian" approach quite out of keeping with a site like Tharros, which is clearly distinguished by quite particular techniques and materials.

This brief review of the techniques and materials considered inappropriate in this particular situation leads to the conclusion that the most suitable approach is one which may be defined as "soft" and which is based as far as possible on the following criteria: reversibility of interventions, clarity of interpretation, and harmonization with the ancient techniques and with the environmental context.

SPECIFICATION OF THE SITUATIONS TO WHICH THE INTERVENTION IS INTENDED TO RESPOND

The specific situations which have been identified as in need of conservation and restoration, and which require different technological responses, can be summarized as follows:

A) Counterscarp wall with revetment in basalt polygonal blocks still in situ, out-of-plumb and shored up in some sections.

B) Inner curtain wall, dry-stone-built with revetment on both faces, in conditions of non-precarious static equilibrium.

C) Counterscarp wall and inner curtain wall in those sections completely denuded of revetment with the consequent exposure of the inner disintegrated rubble core.

OPERATIONAL CHOICES

As regards the counterscarp wall, the need was recognized to reconstitute the original resistant function of the revetment in polygonal blocks to enable it to contain both the inner rubble core on which it abuts and the pressure of the mass of earth comprised by the escarpment to its rear. This means ensuring the cohesion of the revetment's individual basalt blocks with a view to obtaining a continuous and resistant facing.

The chosen operational technique will be exhaustively described in the following report. It should, however, be pointed out in advance that this type of consolidating operation conflicts with the "soft" technology adopted by preference in the proposed interventions as a whole, but is necessitated by static conditions which cannot be satisfied in any other way.

The consolidation of stretches of wall with two revetments *in situ* will be carried out by substantially the same procedure, and the cohesion of the two revetments will be subsequently reinforced by the insertion - to prevent displacement - of suitable tie-beams between them, thus reconstituting the original conditions of containment of the loose stonework interposed between them.

A solution to the conservational problem posed by those stretches of wall in which no revetment has survived is far more complex.

The options of attempting any kind of reconstruction of the revetment, or of impregnating the whole wall mass with liquid cement or epoxide resins, or of consolidating only its outer surface layer, were all rejected.

The assumption was therefore made that the most effective procedure, in spite of its simplicity and cheapness, for achieving the containment of the loose stonework and halting the disintegration and fall of at least the stone components, would consist in the installation of a simple close-mesh net of an appropriate size, and attached to the wall mass it is intended to contain by bolts designed to resist extraction.

To give some idea of the proposed procedure, an analogy may be drawn with the utilization of netting for the containment of motorway embankments. In such cases, a zinc-coated wire net is usually installed; this has the advantages of solidity and rigidity, but requires periodic substitution due to the processes of oxidation. As an alternative the use of a net produced in a synthetic material could be envisaged; this would not require replacement, but would be less rigid.

It is essential, however, that the conservational procedures so far described be accompanied by measures aimed at protecting the structures in question from the damaging effects of erosion by water or wind. In other words, having excluded the options of "direct contact" protection through concrete caps for the tops of the walls, surface treatment with resins for sandstones and cement mortar grout for the joints of the revetments - i.e. for all those structural parts directly exposed to atmospheric agents - it is considered indispensable to take preventive measures to curb the action of the most dangerous factors of climatic and meteorological deterioration, and to do so by operating directly on the causes rather than the effects of such deterioration.

It has therefore been decided to instal a simple protective system consisting of suspended cloths; these will be supported and fastened by a linear framework of metal struts and tie rods, not resting on the archaeological structures. The arrangement and orientation of these cloths will be studied with a view to eliminating contact with rainwater and shielding the impact of the predominant winds and the action of direct sunlight. Their design, based on modular components, will take account of the need for simplicity in installation and dismantling and the easy replacement of damaged parts. Care will also be taken to obtain, both as regards technology utilized and visual impact, a conservational system which is easily removeable and not injurious to the ancient structure. It will further be necessary to ensure the harmonious insertion of this structure in the surrounding landscape, with full respect for its orographical features.

The installation of this protective system based on suspended cloths is judged indispensable at least in those sectors most seriously jeopardized by the visibly accelerating action of climatic and meteorological agents.

Alongside the more incisive static, consolidative and protective measures described above, the implementation of other "soft" treatments extended to the wall structures as a whole is also considered advisable:

- with a view to restoring at least some limited degree of coherence to the inner cores by saturating their cavities and internal discontinuities, recourse has been made to injections of fluid mixtures of clay and sand;

- with a view to compensating for the leaching and erosion of the material lodged in the joints between the polygonal blocks, caused by their recent exposure to the action of climatic and meteorological agents, the wall revetment must be subjected to a thorough process of repair by saturating the lacunae between the joints with the same mixture of sand and earth originally used and still in situ.

MAINTENANCE AND UPKEEP

The provisions, operations and measures hitherto described cannot in themselves constitute a definitive solution to the problem of the conservation of the Tharros monumental complex, nor can their effectiveness be indefinitely prolonged. It is precisely in this case, in fact, that the adoption of a so-called "soft" technology has been decided, since it chiefly makes use of simple and reversible methods and materials which respect the pre-existing structures, and are therefore endowed with a short working life. This is a consequence of the priority choice made to limit the adoption of consolidating materials more resistant than the structures they are intended to consolidate, thus accelerating the latter's rate of disintegration. The limited duration of the deliberately simple measures adopted thus makes the planning of a continuous process of inspection and maintenance indispensable with a view to providing for periodic repairs, adjustments and replacements. This refers in particular to some of the procedures specified above, such as the installation of wire netting, the injection of earth fluids, the supplementation of the filling between joints, and the use of suspended protective cloths.

These specific operations must, of course, be combined with the normal procedures of day-to-day site maintenance. For it is our conviction - which cannot be sufficiently stressed - that an effective conservation policy is not one that is implemented through radical and "definitive" restoration measures, but one that takes the form of a planned sequence of integrated provisions. These consist of: firstly, an indispensable preliminary phase of inspection and the containment of the external causes of deterioration; secondly, a series of limited consolidative measures; and thirdly, a continuous process of periodic maintenance and upkeep.

TITLE: THARROS (OR) : CONSERVATION OF THE PUNIC DITCH. THE OPERATIONAL TECHNIQUE.

AUTHOR: MARIO BELLINI

SUMMARY: Methodological hypothesis of this restoration is that it is necessary to neutralize the causes of damages on the monuments rather than repair their effects. It is proposed, therefore, to program the conservation circumscribing the factors which cause deterioration and proceeding with little maintenance operations within short time intervals.

FOOTNOTE: Engineer, consulant for P.A.T., S.r.l., Rome.

The objective condition of polygonally worked masonry with fine interconnected material delimiting the ditch, as already mentioned in the previous paper, prevented final solutions according to the method set out. Nevertheless, the working method sought for the reinforcement job was not simply made secondary to the stability requirements, but it was desired that it would permit not only the maintenance of the configurations in progress or those to come in the successive excavation, but also that it would respect for the readability of the work in its external facies and its constituent parts.

It was therefore decided to connect the blocks making up the outside faces with steel connectors anchored with epoxy compounds and to install tie-rods that would prevent rotation towards the outside of the faces so connected.

All operations involved in the reinforcement job could be and were carried out by inside lines with the minimum involvement of the visible portions. The work itself was kept clearly separate from the ancient structure so as not to "contaminate" that which emerged from the excavations.

The plan for the work is applicable (with the obvious operational differences) both to the counterscarp wall and the massive wall above delimited by two faces that are also polygonally worked. In fact, the work for the faces is analogous; the tie-rods acting in the counterscarp wall will be anchored in dividers of reinforced concrete, whereas those in the wall above will act between the faces and can be of the passive type.

For the quantitative study of the work we detailed the general information in the previously mentioned publication by Prof. Barreca and that which was gathered by those who had participated in the first excavation program.

It was therefore necessary to limit investigation to the work area only, without being able to further the study of the problems of the general stability of the hill in its western section, the control of the surface water in connection with the presence of fine, easily washed away material on the surface, ~~to~~ the arrangement of the ditch bottom, ~~to~~ the presence of tombs which descend well below the trampled plane.

A series of trial excavations was therefore carried out to determine the below-soil depth of the counterscarp wall and its consistency and internal construction, as well as to learn the characteristics of the soil up against the wall itself.

The wall proved to consist of the visible face for a depth comparable to the maximum dimension of the facade of the single blocks and by a second, coarser face consisting of stony elements of smaller sizes; the enclosed filling was formed of stone fragments and sandy soil.

The soil below the counterscarp wall, which was studied to find the most suitable point for placing the cement dividers to anchor the tie-bars, showed that under a superficial layer of plant soil there is a layer of soil rich in ceramic fragments from various epochs. Beneath this is a thick layer of very pure, absolutely sterile sand of probable eolian deposit,and below this is a layer of life dating back to the Roman age on the basis of the ceramic fragments recovered. a new thin layer of sterile sand, the probable level of Punic life, and, finally, a considerable bank of clay void of findings and having a good resistance.

The anchor dividers were therefore located about 17 meters below the wall in the clay layer. The excavation was carried out by the stratigraphic method under the direction of archeologists from the local authority.

The examination of the trend of the face blocks of the counterscarp wall, which were roughly aligned by diagonal resistent directrices, suggested that these directrices be followed, as best as possible, to created the reconnection between the blocks themselves.

For the boring it was decided to proceed essentially with a sensitive-type, rotation-only bore using diamond bits with the total exclusion of percussion and the consequent vibrations.

The use of rotary-percussion boring with light-weight machines, was limited to that which was necessary to stop the precarious blocks at the top of the wall.

The heads of the holes were located on the side faces, and the union of the blocks was made at about half of their depth and, therefore, at the position of greatest block resistance and with the practical impossibility of being seen from the outside, even in the case of a loss of interconnected material.

The steel rods of the connectors were incorporated into an epoxy compound that was especially formulated to obtain great mechanical resistance, and high thixotropy and adhesiveness to the support.

Injection of epoxy compound was limited to the dimensions of the hole only to avoid the irreversible soaking up of interconnected materials and that which is behind the wall. For this purpose special prefabricated retaining structures were put in even though the thixotropy of the injected material itself gives sufficient guarantee against diffusion.

The face blocks were essentially connected via a single layer, whereas a double layer was used in special cases, such as around the tie-rod heads, to spread out their action better.

For the tie-rods, we used strands of music wire with a polyethylene sheath and slide surfaces of graphitized grease. These were inserted in an additional sheath having a high resistance to mechanical action and chemical agents, and they were further injected with epoxy resin.

The borings were done with continuous core borings with lining of the holes; after insertion of the sheath the covering was removed.

The borings were always begun from the pre-set blocks on the counterscarp wall and went to the anchorage plate of reinforced concrete previously constructed.

The tie-rods were placed under load so that they could immediately exert their effect.

The anchor plates and the blocking parts were placed in recesses made inside the blocks and protected with epoxy mortar. Then the previously bored pieces were replaced in their original positions. The sealing cap was made of powder from the same stone and colored epoxy adhesive. The heads of the seam borings were closed with scabbling from basalt pebbles found in the area.

The work was done in the presence of the temporary wooden structures that were gradually taken down as the reinforcement work proceeded.

With regard to the masonry nuclei and the area in direct contact with the back of the counterscarp wall where internal cavities and discontinuities were found, to give them a minimum consistency, an injection was made of a fluid mixture of sand and clay in the same ratio as that found in situ.

Finally, a general careful check was made of the wall face, saturating the joint spaces with an impasto of soily sand similar to that which is present to this day.

L' OBELISQUE DE PLACE S. GIOVANNI IN LATERANO A ROME

M. GianFranco RUGGIERI*

SUMMARY

The article describes in brief the techniques of intervention used for the consolidation of the Egyptian obelisk situated in the Square of S. Giovanni in Laterano in Rome.

* Architect of the Soprintendenza ai Beni Architettonici ed Ambientali del Lazio

L'OBELISQUE DE PLACE ST-JEAN-DE-LATRAN

L'obélisque, en granit rouge, fut érigé devant le Temple d'Ammon à Thèbes, Egypte, par les Pharaons Thoutmôsis III et Thoutmôsis IV, au XVe siècle av. J.-C. Constance II, fils de Constantin, le transporta à Rome en 357 apr. J.-C. par un navire construit à cet effet. L'obélisque fut dressé au Circus Maximus où il fut retrouvé en trois morceaux en 1587.

En 1588, sur l'ordre du Pape Sixte Quint, l'obélisque fut dressé dans son site actuel par Domenico Fontano.

Cet obélisque, qui mesure 31 mètres (47 m avec sa base), est le plus haut et le plus ancien de Rome.

Il y a déjà quelques années, des ouvrages de consolidation furent accomplis, mais la circulation croissante autour de l'obélisque, qui constitue un sens giratoire pour les voitures, ainsi que la chute d'un certain nombre de ses éléments en granit et en bronze, ont amené la Direction Générale pour la conservation des Beaux-Arts à effectuer une nouvelle intervention plus radicale.

On a réalisé tout d'abord des tests sur ses fondations en vue d'étudier leur structure et leur état de conservation.

Ces fondations comportent des blocs de travertin capables de supporter le poids supérieur qui se situe à quelque 10.000 quintaux. Des infiltrations d'eau, dues au fonctionnement défaillant de l'installation de décharge de la fontaine située à la base de l'obélisque, ont été éliminées.

Le soubassement de travertin n'a montré la moindre lézarde. Dès lors, l'attention s'est concentrée sur l'obélisque lui-même.

On sait qu'il fut retrouvé en trois morceaux et que Domenico Fontana le dressa deux ans après avoir dressé celui de Place St.-Pierre; mais à l'heure actuelle, il ressort que les morceaux sont plus de trois.

Les divers éléments furent ancrés par Fontana à l'aide de crampons de fer noyés dans le plomb, en laissant de grans espaces ouverts entre chaque bloc.

Un examen minutieux du monument a révélé la présence de craquelages dans le granit, notamment à un tiers de son hauteur vers le haut. En outre, quelques blocs présentaient un phénomène modeste de délitement de l'écorce superficielle de la pierre; vraisemblablement ce phénomène tient au premier chef aux agents atmosphériques et aux vibrations.

Au cours de l'étude on a trouvé quelques crampons cassés qui présentaient également les traces de l'usure et d'un énervement de traction léger, tandis que l'ancrage d'autres crampons était sorti du logement de plomb.

Au sommet, le faîte de bronze, comportant quatre lions rampants avec des montagnes au-dessus (l'écusson de Sixte Quint) surmontés d'une grande croix, présentait les traces évidentes d'une détérioration due aux agents atmosphériques.

Le travail a impliqué tout d'abord le démontage du faîte qui a été descendu et placé en bas.

Les armatures de fer du faîte étaient oxydées et corrodées à tel point que leur soutien n'était plus fiable; par conséquent elles ont été enlevées et remplacées par de nouvelles barres en acier inoxydable placées dans le granit.

Le faîte a été transporté au laboratoire où les rivets de cuivre cassés ont été remplacés, et de nouveaux rivets ont été montés où ils faisaient défaut.

De plus, on a bosselé quelques petits morceaux en cuivre qui n'existaient plus ou étaient usés, en vue de compléter l'ensemble et d'empêcher les oiseaux de nicher dans les espaces vides.

Cet ensemble a été monté à nouveau au moyen de ligatures en cuivre bien lié aux nouvelles armatures en acier inoxydable.

Aucun traitement particulier n'a été envisagé sur le bronze, au moyen de substances comportant des silicones.

Pour ce qui est de l'obélisque, on a décidé de remettre en état ou d'améliorer sa capacité de supporter les contraintes de traction auxquelles les divers blocs étaient soumis, tant à cause des vibrations et des oscillations dues à la circulation, qu'à cause de la fonction statique réduite des crampons originaires qui ont été convenablement placés dans les logements de plomb.

Pour pouvoir réaliser cette intervention, on a effectué des forages à l'aide de pointes de diamant à circulation d'eau qui traversent transversalement les blocs contigus.

Dans les trous ainsi exécutés on a installé des barres d'acier inoxydable à haute limite d'élasticité - capables de supporter des tractions jusqu'à 25 tonnes - jointes aux blocs qu'elles traversent au niveau de la tête et du pied, grâce à des cartouches spéciales de résine époxyde. Les têtes des entraits sont légèrement encaissées par rapport au plan extérieur du granit.

Un problème particulier a été celui de fermer les différents espaces vides existant entre les blocs, à l'exclusion bien entendu des trous carrés qu'utilisaient les Egyptiens pour soulever l'obélisque au moyen d'échafaudages appropriés.

Etant donné que ces espaces vides étaient aisément accessibles aux oiseaux pour leur nidification, et compte tenu des dégâts provoqués par les infiltrations d'eau météorique qui sont souvent soumises, en dépit de l'hiver doux de Rome, au gel et au dégel, on a jugé opportun de fermer ces espaces vides.

Cette fermeture, qui a été d'abord effectuée à titre expérimental entre deux blocs avec de la résine époxyde, a été contrôlée pendant une période d'environ trois mois.

A la fin de cette période, peut-être à cause de l'épaisseur du scellement ou par réaction avec le granit ou encore à cause de la qualité de la résine, cette fermeture avait changé de couleur, en passant d'une couleur neutre à une couleur vert clair que l'on pouvait observer distinctement même à distance.

Dès lors, les espaces vides n'ont été scellés avec des résines qu'en profondeur, alors que dans la zone superficielle on a réalisé des masticages comportant des grenailles de coulis et de la poudre de granit.

L'obélisque a été finalement brossé et lavé avec de l'eau et de l'acide acétique afin d'éliminer tant les incrustations calcaires que les incrustations dues à l'écoulement noirâtre, dû à son tour à la présence des crampons de fer.

Le soubassement de travertin a subi également un traitement similaire; en outre on a assuré l'imperméabilisation de sa petite corniche au moyen d'une gaine élastométrique en vue d'empêcher les eaux météoriques de s'infiltrer.

De plus, une nouvelle couche de la peinture noire traditionnelle a été repassé sur les lettres des inscriptions, qui désormais n'étaient plus lisibles.

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De plus, une nouvelle couche de terrain

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l'obélisque au moyen

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par les infiltrations

Section 5 :

Study and analysis of ancient mortars and renderings.

Etude et analyse des mortiers et enduits anciens.

L'ANALYSE DES MORTIERS ET ENDUITS DES PEINTURES MURALES ET DES BATIMENTS ANCIENS

Dupas Michel

SUMMARY

In this paper, the author gives a method for the analysis of mortars in monuments and wall paintings. The procedure requires an extraction of soluble salts in water, an attack by hydrochloric acid, a calcination at 900°C and a determination of the CO_2 content. The purposes of this analysis are described.

The procedure has the advantage to enable the determination of hydraulic silicates, to distinguish the different kinds of limes and to give instructions on the state of conservation of mortars.

The analytical methods do not need any expensive material.

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24 juillet 1981

I INTRODUCTION

L'analyse des mortiers et enduits constitue une des tâches principales d'un laboratoire ayant pour objectifs l'étude et la conservation des bâtiments anciens. Dans le cadre des recherches de l'Institut royal du Patrimoine artistique, nous nous sommes attachés à mettre au point un mode opératoire d'analyse des mortiers qui puisse apporter des enseignements sur leur état de conservation et leur composition, et permette de caractériser les divers types de chaux utilisées.

Le mode opératoire proposé peut être appliqué, par extension, à l'analyse des matériaux pierreux.

En ce qui concerne les méthodes analytiques, notre choix s'est porté sur la titrimétrie, la colorimétrie, la spectrophotométrie d'émission dans la flamme et la spectrophotométrie d'absorption atomique. Ces deux dernières méthodes sont à conseiller pour cette étude.

Quelques éléments seulement ont pu être dosés par ces techniques modernes d'analyse, notre dispositif à flamme étant peu performant. Cet exposé montre qu'on peut obtenir de bons résultats en faisant appel aux méthodes classiques quand on ne dispose pas d'un tel équipement.

II BUT DE L'ANALYSE DES MORTIERS ET ENDUITS

L'analyse des mortiers et enduits anciens est réalisée dans le but de fournir des enseignements d'ordre historique, technologique ou scientifique dans les domaines suivants :

1°) la restauration des monuments : si l'analyse montre que le mortier est en bon état de conservation, on peut utiliser pour la restauration un mortier analogue ou de composition voisine qui soit en harmonie avec la construction ancienne et satisfasse aux exigences de celle-ci. Dans le cas contraire, une étude plus complète s'impose pour connaître les raisons pour lesquelles le matériau n'a pu remplir son rôle efficacement : rapport liant-sable mal établi, altération par les sels, etc...

2°) l'étude des sites archéologiques : la connaissance de la composition des mortiers présente un intérêt historique et scientifique notamment lorsqu'on se trouve en présence de mortiers datés.

3°) l'étude des relations entre la composition des mortiers et la chronologie des constructions : l'utilité des analyses dans ce domaine a été établie dans l'étude des monuments historiques de Pologne (Jedrzejewska, 1967). Elle ne paraît cependant pas évidente en ce qui concerne nos monuments ; dans de nombreux cas, en effet, une trop grande dispersion dans les résultats ne permet pas un classement par rapport à des mortiers datés.

4°) l'étude des peintures murales : l'analyse des enduits est réalisée dans le but de comparer les peintures et leurs supports, d'étudier leurs altérations ou d'apporter des enseignements d'ordre technologique en fonction de l'usage des lieux et de l'époque. L'étude des relations entre la chronologie des oeuvres et la composition des mortiers pourrait être étendue aux mortiers de peintures murales, ces derniers étant plus homogènes que ceux des bâtiments anciens.

III LES PROCEDES D'ANALYSE DES MORTIERS

Le relevé de la bibliographie montre que les mortiers peuvent être analysés de diverses manières. Les procédés utilisés sont ceux que l'on applique aux matériaux silicatés. Parmi les plus courants, on retient la fusion en présence de carbonate sodicopotassique et l'attaque par l'acide chlorhydrique.

Dans le procédé par fusion, la matière est fondue en présence d'environ dix fois son poids de carbonate sodicopotassique. Après refroidissement, le produit de la fusion est repris par l'acide chlorhydrique dilué et évaporé à sec. L'insoluble est constitué uniquement de silice. Ce procédé ne peut être retenu car il ne met pas en évidence les diverses variétés de silice rencontrées dans les mortiers, en particulier la silice constitutive des silicates et silicoaluminates hydrauliques.

Les méthodes utilisant l'acide chlorhydrique pour décomposer les mortiers sont les plus intéressantes. L'attaque de la matière peut s'effectuer à chaud ou à froid.

Dans le procédé à chaud, la matière d'abord délayée dans l'eau est attaquée par l'acide chlorhydrique dilué au demi (HCl 1:1) de façon à obtenir une concentration en HCl de 10% en volume. On chauffe rapidement jusqu'à ce que la solution prenne une teinte jaune clair. On filtre immédiatement à chaud et lave à l'eau bouillante. Le filtre et son contenu sont ensuite traités à ébullition par une solution à 50g par litre de carbonate de sodium. Après filtration et lavages, l'insoluble est calciné et pesé. Cet insoluble est alors fondu en présence de carbonate sodicopotassique. Le produit de la fusion repris par HCl dilué et évaporé à sec permet de doser la silice insoluble. Une fusion analogue, réalisée sur une autre aliquote de matière, permet de doser la silice totale et tous les cations à l'exception des alcalins. La silice soluble, constitutive des composés hydrauliques, est obtenue par différence entre la silice totale et la silice insoluble. (Voinovitch— Debras - Guédon— Louvrier, 1962)

Ce procédé est digne d'intérêt car il caractérise les diverses variétés de silice. Il est très précis mais long à réaliser. Le dosage de la silice insoluble nécessite en effet deux solubilisations dans HCl, une dans le carbonate de sodium, de nombreux lavages, deux calcinations et deux fusions alcalines.

Dans les procédés à froid, la matière est mise en présence d'HCl dilué pendant un temps relativement long. Le quartz et les silicates de fer et d'aluminium constitutifs du sable ne sont pas décomposés et peuvent être dosés. Ils représentent l'insoluble. La silice constitutive des silicates hydrauliques n'est totalement solubilisée que dans certaines conditions qu'il est nécessaire de déterminer.

H. Jedrzejewska a effectué en Pologne une série impressionnante d'analyses de mortiers anciens dans le but d'étudier les relations entre la chronologie des constructions et la composition des mortiers. Son procédé permet la classification des mortiers à partir de trois déterminations : les teneurs en anhydride carbonique, en sable et en matières solubles. L'attaque de la matière est effectuée avec HCl 1:1. Le volume de CO_2 dégagé est exprimé en $CaCO_3$ et l'insoluble représente le sable. La teneur en matières solubles représente la différence entre 100% et la somme des deux autres constituants (Jedrzejewska, 1960). En fonction des résultats obtenus, il est intéressant de constater qu'il n'est pas toujours nécessaire de réaliser une analyse complète du matériau. Un problème posé d'une façon précise oriente bien souvent l'analyse chimique vers la détermination de quelques constituants seulement. Ce procédé d'analyse des mortiers ne tient cependant pas compte de la présence éventuelle de carbonate de magnésium et de sels solubles dans l'eau. Il ne met pas en évidence les diverses variétés de silice et ne permet donc pas de caractériser le type de chaux utilisée dans la préparation du mortier.

Après examen des diverses méthodes, nous nous sommes attachés à mettre au point un procédé d'analyse des mortiers anciens à partir d'une attaque de la matière par HCl et nous avons essayé de définir les conditions opératoires qui permettent de solubiliser complètement la silice constitutive des silicates hydrauliques. Le dosage de cette silice, réalisé par insolubilisation à partir d'une aliquote du filtrat chlorhydrique, devrait mettre en évidence la présence de silicates hydrauliques et permettrait de caractériser les divers types de chaux utilisées dans la préparation des mortiers.

IV CONDITIONS DE SOLUBILISATION DES SILICATES HYDRAULIQUES

Essais de laboratoire

a. Solubilité du kaolin dans HCl à 20% en volume (HCl 1:4)

Le kaolin est constitué en majeure partie de kaolinite, silicate hydraté d'alumine de composition $Al_2O_3 . 2\ SiO_2 .\ 2H_2O$. La kaolinite est le constituant principal des argiles. On la rencontre donc dans les sables argileux. Les essais réalisés montrent que 0,57% de silice passe en solution lorsque l'attaque est effectuée pendant une heure au bain-marie bouillant. A température ambiante, l'acide ne solubilise que 0,08% de silice. Il est donc recommandé de travailler à température ambiante pour empêcher la décomposition des sables argileux.

b. Solubilité du silicate de sodium dans HCl 1:4.

Les essais ont été effectués dans le but d'observer le comportement de solutions alcalines riches en silice et mises en présence d'HCl dilué. Dans des béchers de 150ml, on a introduit respectivement 5, 10, 20, 30, 40 et 50ml d'une solution de silicate de sodium titrant 40mg de SiO_2 par ml. On a ajouté ensuite dans chaque bécher 20ml d'HCl 1:4 et dilué les solutions à 100ml avec de l'eau déminéralisée. Les solutions homogénéisées ont été conservées à température ambiante. Nous avons remarqué après dix jours la formation d'un gel de silice dans la solution la plus concentrée. Quelques essais complémentaires réalisés sur des solutions plus concentrées en silicate de sodium montrent une gélification plus rapide dans les mêmes conditions. Les résultats obtenus prouvent qu'on peut maintenir en solution pendant plusieursjours de la silice en milieu HCl très dilué lorsque cette teneur en silice est inférieure à 2g dans 100ml de solution acide.

c. Mise en solution des constituants hydrauliques d'un ciment.

Les essais ont été réalisés dans le but d'étudier le comportement des silicates et aluminosilicates de calcium en présence d'acide chlorhydrique. Ces composés riches en silice combinée se trouvent associés aux aluminates de calcium dans les liants hydrauliques. Nous avons successivement examiné l'influence de la concentration de l'acide chlorhydrique, du temps de réaction et de la température sur la solubilisation de la silice combinée. Les résultats des essais tendent à montrer que :

1- une augmentation de la concentration de l'acide chlorhydrique favorise la formation de silice colloïdale. Sous cette forme la silice reste soluble mais ne réagit pas avec le molybdate d'ammonium. Le dosage de la silice solubilisée doit être effectué par gravimétrie après évaporation à sec d'une aliquote du filtrat chlorhydrique.

2- pour des concentrations en HCl comprises entre 20 et 50% en volume, la quantité de silice mise en solution reste à peu près constante.

3- la quantité de silice solubilisée diminue légèrement quand le temps de réaction augmente. L'acide chlorhydrique à 20% en volume réagissant avec le ciment pendant 3, 16 et 24 heures solubilise respectivement 15,5 - 14,9 et 14,6% de silice.

4- la quantité de silice solubilisée diminue fortement quand l'attaque du ciment est réalisée à chaud. Elle passe de 15,5 à 10% lorsque l'attaque est effectuée par HCl 1:4 pendant une heure au bain-marie bouillant.

Conclusions

Pour solubiliser au maximum la silice constitutive des composés hydrauliques, il faut :

1- réaliser l'attaque avec de l'acide chlorhydrique très dilué afin de favoriser la mise en solution des divers composants et limiter la formation de silice colloïdale.

2- travailler à température ambiante pour éviter la précipitation d'une partie de la silice combinée.

3- limiter le temps de réaction.

En fonction de ces considérations, la mise en solution des mortiers et enduits anciens sera effectuée à température ambiante en présence d'acide chlorhydrique à 20% en volume. Le temps de réaction sera fixé à 16 heures.

Remarques :

1- Les essais ont montré qu'une attaque effectuée sur un ciment par HCl 1:4 pendant 3 heures permet de solubiliser au maximum la silice combinée. Nous avons cependant adopté un temps de réaction de 16 heures dans le but d'assurer la mise en solution complète des oxydes de fer et d'aluminium.

2- Le dosage de la silice solubilisée doit être effectué par gravimétrie après évaporation à sec d'une aliquote du filtrat chlorhydrique.

V L'ANALYSE DES MORTIERS ET ENDUITS ANCIENS METHODE PROPOSEE

Echantillonnage

L'hétérogénéité des mortiers anciens rend le problème de l'échantillonnage particulièrement délicat. Cette opération très importante doit être effectuée avec le plus grand soin pour que les résultats puissent être significatifs. Les prélèvements d'échantillons sont régis selon des normes bien établies ; malgré cela on constate que les laboratoires reçoivent encore des prélèvements isolés, mal localisés et non représentatifs. Ces échantillons n'ont aucune valeur scientifique et les résultats d'examens permettent à peine d'approcher le problème concerné.

En vue d'étudier les relations entre la composition des mortiers et la chronologie des monuments de Pologne, H. Jedrzejewska a défini les conditions à respecter pour rendre les prélèvements de mortiers significatifs dans une note technique qui constitue une référence précieuse pour l'analyste. (Jedrzejewska, 1976)

Méthodologie

L'échantillon, dont le poids se situe entre 100 et 500g selon son degré d'hétérogénéité, subit d'abord un concassage afin de le débarrasser de sa charge. Dans les mortiers de fouilles archéologiques, la charge peut être constituée de pierrailles, calcaires ou non, de galets, de déchets de briques ou encore de restes de bois utilisés pour la calcination des pierres à chaux. Dans les enduits de peintures murales, cette charge, généralement moins importante, peut être constituée de crins ou de paille.
Les mortiers de peintures murales romaines peuvent contenir du marbre broyé destiné à lisser la préparation et à apporter une note colorée à la composition. Dans la mesure du possible, la charge doit être séparée du mortier de façon à ne pas modifier le rapport sable-liant initial. Son identification peut apporter des enseignements sur la technologie des oeuvres.

Débarrassé de sa charge, le mortier est ensuite broyé, passé au tamis de 250 microns et séché à 105°C jusqu'à poids constant.

L'analyse est réalisée à partir de quatre aliquotes.

Sur la 1e aliquote (± 2g) on réalise l'extraction des sels solubles dans l'eau. L'opération s'effectue à température ambiante avec de l'eau déminéralisée bouillie et la mise en solution des sels est assurée grâce à l'action énergique d'un secoueur. L'extrait filtré permet de doser les ions SO_4^{-2}, Cl^-, NO_2^-, NO_3^-, Ca^{+2}, Mg^{+2}, Na^+ et K^+. L'examen des sels solubles permet de juger l'état de conservation du mortier. (Dupas, 1971)

Sur la 2e aliquote (1g) on réalise à température ambiante une attaque chlorhydrique au moyen de 100ml d'HCl 1:4 et laisse réagir pendant 16 heures. L'insoluble, constitué de silice, de silicates de fer et d'alumine et éventuellement de matières organiques, est calciné à 900°C et pesé. Le filtrat amené à 500ml permet de doser les ions Ca^{+2}, Mg^{+2}, Fe^{+3}, Al^{+3}, Na^+ et K^+. Ce procédé présente l'avantage de laisser en solution la silice des composés hydrauliques. Elle peut être dosée et attribuée correctement à la fraction "liant" du mortier. Par contre une attaque par HCl chaud provoquerait une insolubilisation partielle de cette silice que l'on attribuerait à tort à la fraction "sable".

Sur la 3e aliquote (± 1g) on détermine la perte au feu (P.F.) à 900°C. Elle correspond à la somme des teneurs en CO_2, eau des argiles $(H_2O)_a$, matières organiques (M.O.) et eau des sels $(H_2O)_s$.

La 4e aliquote (0,4 à 1g) est destinée au dosage du CO_2 capté par la chaux du mortier.

<u>Expression des résultats</u>. <u>Analyse rationnelle</u>

L'analyse rationnelle est calculée à partir des résultats d'analyse de l'extrait aqueux et du filtrat chlorhydrique. Ce calcul peut être facilité grâce à un examen de la matière par diffraction des rayons X qui permet d'en identifier les divers constituants.

a. Analyse rationnelle de l'extrait aqueux.

En fonction des affinités des éléments en présence et de notre expérience, il semble qu'on puisse l'établir comme suit :

1°) exprimer les teneurs en cations et en anions en milliéquivalents grammes.

2°) attribuer les ions SO_4^{-2} aux ions Ca^{+2} et exprimer le sel obtenu en $CaSO_4 2H_2O$. L'excès d'ions SO_4^{-2} est attribué aux ions K^+ et exprimé en K_2SO_4. En leur absence, cet excès est exprimé en Na_2SO_4.

3°) attribuer les ions Cl^- aux ions K^+ et exprimer le sel obtenu en KCl. En l'absence d'ions K^+, on exprime en Na Cl.

4°) attribuer les ions NO_3^- aux ions Mg^{+2} et exprimer le sel obtenu en $Mg (NO_3)_2$. En l'absence d'ions Mg^{+2}, on exprime en $NaNO_3$.

Remarques : - les teneurs en ions NO_2^- rencontrées dans nos échantillons sont généralement négligeables ; c'est pourquoi nous n'en tenons pas compte dans le calcul de l'analyse rationnelle de l'extrait aqueux.

- l'analyse rationnelle met en évidence la présence de sels dangereux pour la conservation des matériaux pierreux. Le sulfate de sodium et le nitrate de magnésium s'hydratent et se déshydratent selon les conditions de température et d'hygrométrie et les tensions développées par ces changements d'états disloquent peu à peu la surface du matériau.

b. Analyse rationnelle du filtrat chlorhydrique.

Ce filtrat contient les constituants de l'extrait aqueux et d'autres dont l'analyse rationnelle peut être établie comme suit :

1°) exprimer les teneurs en cations en milliéquivalents grammes et retrancher de ces valeurs celles correspondant à l'extrait aqueux.

2°) exprimer les ions Mg^{+2} en $Mg CO_3$ et calculer la teneur en CO_2 attribuée.

3°) retrancher de la teneur en CO_2 de l'échantillon celle sous forme de $MgCO_3$, exprimer cet excédent en $CaCO_3$ et calculer la teneur en ions Ca^{+2} attribuée.

4°) exprimer l'excédent d'ions Ca^{+2} en CaO. La valeur obtenue correspond à la teneur en CaO des composés hydrauliques.

5°) exprimer les ions Fe^{+3}, Al^{+3}, Na^+, K^+ et Si^{+4} en oxydes. Liés au CaO ils constituent les composés hydrauliques.

C. Composition de l'échantillon.

Elle s'exprime en % en poids. Aux constituants trouvés en a et b, il convient d'ajouter la teneur en insoluble, l'eau des silico-aluminates $(H_2O)_a$ et les matières organiques (M.O.). La somme $(H_2O)_a$ + (M.O.) est calculée à partir de (P.F.), de (CO_2) et de $(H_2O)_s$. On sait en effet que :

$(P.F.) = (CO_2) + (H_2O)_s + (H_2O)_a + (M.O.)$
$(H_2O)_s$ est calculée à partir des teneurs en sels de l'extrait aqueux.

La connaissance des teneurs en SiO_2 combinée, Fe_2O_3, Al_2O_3, CaO et MgO permet de calculer l'indice d'hydraulicité de la chaux utilisée pour la préparation du mortier et de la classer dans l'échelle des chaux hydrauliques.

VI DESCRIPTION DES METHODES ANALYTIQUES

Dosage des sulfates (Dupas, 1968)

Les sulfates sont dosés par titrimétrie au moyen d'une solution de perchlorate de baryum 0,005 M en présence de thorin comme indicateur. Les cations sont éliminés par passage sur une résine d'échange cationique fortement acide du type H^+. Le dosage se réalise en milieu isopropanol à 80%, à un pH compris entre 2,5 et 4,0 que l'on ajuste au moyen d'acide perchlorique à 10%. Les phosphates gênent. La présence des nitrates n'est gênante qu'à partir d'une concentration égale à la moitié de celle des sulfates. La méthode permet de doser 0,10 à 5mg d'ions sulfates dans 10ml de solution traitée. Le terme du dosage se manifeste par le virage de l'indicateur du jaune au rose. La méthode est très reproductible et convient à la microanalyse.

Dosage des chlorures (Charlot, 1966)

Le dosage des chlorures s'effectue par titrimétrie au moyen d'une solution de nitrate mercurique 0,02 N. A la solution contenant jusque 5mg d'ions Cl^-, on ajoute 5 gouttes de bleu de bromophénol à 0,05% dans l'alcool, neutralise par HNO_3 0,05 N si l'indicateur est bleu ou par NaOH 0,05 N s'il est jaune. A neutralité, on ajoute 1ml d'acide en excès, dilue à 100 ml et ajoute 5 gouttes de diphénylcarbazone à 0,5% dans l'alcool. On titre avec le nitrate mercurique jusqu'à virage du jaune pâle au violet. Les bromures sont titrés en même temps.

Dosage des nitrites (Dupas, 1968)

Présents en concentration généralement infime, les nitrites sont dosés par colorimétrie par le réactif de Griess à base d'acide sulfanilique et de 1- naphtylamine en milieu acétique. La coloration rose se développe à un pH compris entre 1,7 et 3,0 et les mesures sont effectuées après une heure d'attente pour obtenir une coloration stable. Les mesures s'effectuent à 520 nm. On dose au maximum 0,3 mg d'ion NO_2^- par litre. Les ions Fe^{+3} doivent être complexés par l'acide citrique.

Dosage des nitrates (Charlot, 1966)

Le dosage colorimétrique des nitrates s'effectue en l'absence de chlorures. On les élimine par centrifugation après précipitation au moyen de la quantité juste équivalente de sulfate d'argent. La solution exempte d'ions Cl^- et Ag^+ est additionnée de 10 gouttes d'acide acétique à 20% et de perhydrol pour décomposer les carbonates et oxyder les nitrites. On évapore la solution à sec et, après refroidissement, ajoute 1ml d'acide phénoldisulfonique et laisse réagir pendant 10 minutes. On dilue et ajoute 10ml d'ammoniaque 1:1. Les solutions prennent une coloration jaune en présence de nitrates. On amène ensuite à 50ml et effectue la colorimétrie à 410nm. On dose au maximum 0,4mg d'ion NO_3^- dans la fiole de 50ml.

Dosage du calcium (Merck, 3e éd.)

Le dosage du calcium se réalise directement par complexométrie au moyen d'une solution de complexon III 0,02M en présence d'un indicateur à base de calcéine, utilisé en trituration avec la thymolphtaléine et le nitrate de potassium. Les ions Mg^{+2} ne gênent pas. L'effet perturbateur des ions Fe^{+3}, Al^{+3} et Mn^{+2} est annihilé par la triéthanolamine. La solution est amenée à pH 12 par addition de KOH 2N, on ajoute ensuite environ 20mg d'indicateur et titre avec le complexon III jusqu'à disparition de la fluorescence verte et apparition d'une coloration violette stable. La méthode est rapide et précise.

Dosage du magnésium

a. Par spectrophotométrie d'absorption atomique. (Firman, 1965)

On dresse une droite d'étalonnage pour des concentrations en Mg^{+2} comprises entre 0,2 et 2,0 ppm. Les mesures sont effectuées à 285,2 nm en présence de 200 ppm de Ca, vu la forte concentration de cet élément dans nos échantillons, et de 2500 ppm de Sr pour éliminer la plupart des interférences. L'atomisation est réalisée dans la flamme air-acétylène. Le coefficient angulaire de la droite d'étalonnage augmente avec le débit d'acétylène. Pour obtenir des résultats très précis, on calcule les concentrations par la méthode d'encadrement au moyen de deux étalons. Cette méthode convient parfaitement au dosage des faibles teneurs en magnésium dans les matériaux riches en calcium.

b. Par complexométrie (Merck, 3e éd.)

La somme Ca + Mg est dosée par complexométrie par le complexon III 0,02 M. Connaissant la teneur en calcium, on trouve celle en magnésium par différence. La triéthanolamine élimine l'effet perturbateur des ions Fe^{+3}, Al^{+3} et Mn^{+2}. Après addition de la triéthanolamine, la solution doit être amenée à pH 4 au moyen de soude diluée. On ajoute ensuite 1 comprimé tampon indicateur "Merck", 1ml d'ammoniaque et titre avec le complexon III jusqu'à virage de l'indicateur du rouge au vert. Ce dosage n'est pas à conseiller quand le rapport Ca/Mg est très élevé.

Dosage du fer. (Voinovitch – Debras-Guédon – Louvrier, 1962)

Le dosage colorimétrique des ions Fe^{+3} s'effectue au moyen d'acide sulfosalicylique. Dans une fiole de 100 ml, on introduit une aliquote contenant au maximum 1mg de Fe_2O_3. On ajoute dans l'ordre 5ml de NH_4Cl à 54g/l, 10ml d'acide sulfosalicylique à 12% et de l'ammoniaque 1:3, goutte à goutte, jusqu'à virage de la solution au jaune vif. On ajoute ensuite 1ml d'ammoniaque 1:3 en excès et amène à 100ml. La colorimétrie s'effectue à 420nm. Les constituants habituels des matériaux pierreux ne gênent pas.

Dosage de l'aluminium

a. Par complexométrie. (Voinovitch – Debras-Guédon – Louvrier, 1962)

Ce dosage s'applique lorsque la prise d'essai contient au moins 0,3 mg d'Al^{+3}. Les ions Fe^{+3} et Ti^{+3} gênent et sont extraits par le chloroforme sous forme de cupferronates. A la solution traitée, on ajoute du complexon III 0,02 M en excès et on titre en retour cet excès au moyen d'une solution de sulfate de zinc 0,02M. Le dosage est réalisé à pH 4,5 en présence de 50% d'alcool. L'indicateur est le dithizone. Au terme du virage, il vire du violet au rouge franc.

b. Par colorimétrie (Hill, 1956)

Les ions Al^{+3} sont dosés par colorimétrie au moyen d'ériochrome cyanine R. Le fer gêne la réaction. Pour éliminer son influence on réalise le dosage à partir de deux aliquotes identiques, en fioles de 50 ml. Dans la fiole témoin, le fer et l'aluminium sont complexés par le complexon III ; dans la fiole de mesure, le fer seul est complexé par l'acide thioglycolique. L'ériochrome cyanine ne peut réagir qu'avec l'aluminium car le fer est complexé dans les deux fioles. La méthode est très sensible ; on dose en effet au maximum 0,03mg d'aluminium dans la prise d'essai. La colorimétrie s'effectue à 535 nm.

Dosage du sodium ou du potassium. (Merck, note technique)

Le dosage est réalisé par spectrophotométrie d'émission dans la flamme air-acétylène. Il s'agit de la méthode de Schinkel et Schuhknecht permettant le dosage des ions Na^+, K^+ et Li^+. On dresse une droite d'étalonnage à partir de solutions contenant 0,25 à 2ppm de sodium. Les mesures de transmission sont effectuées à 589,3 nm en présence de 200ppm de Ca et 10ml de tampon à base de nitrate d'aluminium et de chlorure de césium. Le calcul des concentrations s'effectue avec précision par la méthode d'encadrement au moyen de deux étalons. Pour doser le potassium, on effectue les mesuresà 768,2 nm.

Dosage de la silice solubilisée.

La silice solubilisée constitutive des silicates hydrauliques est dosée par gravimétrie. La silice est insolubilisée en milieu HCl par évaporation à sec d'une aliquote du filtrat chlorhydrique. La silice recueillie est lavée, calcinée à 900°C, refroidie et pesée.

Dosage de l'anhydride carbonique. (Dupas, 1971)

Le dosage volumétrique du CO_2 se réalise dans l'appareil de Baur-Cramer. Dans un erlenmeyer de 100ml de capacité, on introduit un poids connu de matière et un godet contenant HCl 1:1. Le bouchon est relié à une burette remplie d'une solution de $CuSO_4$-H_2SO_4. On lit le volume de CO_2 dégagé lors du contact acide-carbonate. On encadre par deux mesures sur $CaCO_3$ étalon, détermine l'équivalent par ml et calcule la teneur en CO_2 de la matière. On doit travailler rapidement pour éviter l'influence des variations de température et de pression.

VII CONSIDERATIONS GENERALES

Cet exposé apporte une contribution à l'étude des problèmes de conservation des monuments. Le mode opératoire décrit pour analyser les mortiers peut s'appliquer aux matériaux pierreux. Il met en évidence la caractérisation des diverses variétés de silice et permet de différencier les types de chaux utilisées dans les mortiers.

Ce procédé, appliqué à l'Institut royal du Patrimoine artistique depuis plusieurs années déjà, nous permet d'intervenir dans les problèmes de restauration des monuments, d'étudier la technologie des peintures murales et de rechercher les relations existant entre la composition des mortiers et la chronologie des constructions.

Les méthodes de dosages décrites dans cet exposé ne nécessitent pas un appareillage coûteux et peuvent donc être appliquées dans la plupart des laboratoires.

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TITLE: CHEMICAL TECHNIQUES OF HISTORIC MORTAR ANALYSIS

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SUMMARY

Eight mortar standards of known composition were prepared and used to test the accuracy and applicability of three chemical techniques widely used to analyze historic mortars. The results indicated that only one of these techniques can be considered reliable when used to examine a broad range of historic mortars.

FOOTNOTE

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INTRODUCTION

Before using any new technique it is common practice in analytical work to evaluate it using standard samples of known composition. Only in this way is it possible to determine the applicability and accuracy of the technique. In fact, it is normal practice when publishing a new analytical scheme to also publish the results of such standard analyses. However, when dealing with the analyses of historical materials it is often difficult to obtain suitable standards; thus, unfortunately this necessary empirical proof of a technique's reliability is often lacking.

In a desire to find the advantages and limitations of three commonly used and well known procedures for the analysis of historic mortars, eight mortars of known composition were prepared and cured in the laboratory. These were chosen to represent types of 19th and early 20th Century mortars found in Canada and which would test the limits of the analytical schemes but be neither unusual nor exotic. These are not historic mortars, yet the results for these modern standards will be a measure of the applicability and accuracy of the examined techniques.

The three techniques studied are those of H. Jedrzejewska (Jedrzejewska, 1960), E.B. Cliver (Cliver, 1974) and the American Society for Testing and Materials (American Society for Testing and Materials, Designation C85-66, 1971).

The technique of H. Jedrzejewska is a simplified semi-quantitative procedure designed to classify large numbers of samples to aid in comparitive studies and in dating mortars. It was not intended to be used as a technique to determine the type and proportions of all ingredients; however, we wished to find out if it could be used reliably as a rapid screening test to distinguish between lime mortars and mortars which may possess hydraulic properties.

The technique determines three properties of mortars: by volumetric analysis the carbon dioxide content which is mathematically converted to the calcium carbonate content; by gravimetry the sand content; and by difference the content of complex silicates (soluble fraction). It is the last value which is of primary interest in a screening test; a high content of complex silicates would indicate an hydraulic component and a low content would indicate a non-hydraulic lime mortar.

The technique of E.B. Cliver is designed as a simple, relatively accurate technique to determine the nature and amount of components used in mixing mortars. By gravimetry three values are determined:

1) The soluble fraction (lime and Portland cement solubles)
2) The sand fraction
3) The fine residue fraction

In this test the type of mortar is determined by the colour of the fine residue fraction. If the fine residue is medium to dark grey then Portland cement was used in the mix. Mr. Cliver assumes that 40% of Portland cement is acid insoluble, thus knowing the amount of fine residue present the amount of Portland cement in the sample can be calculated.

If the fine residue is reddish to light tan in colour then it results from clay in the mix. If the residue is brown and at levels approximating the lime content then a natural cement was used. In the latter two cases further calculations based on the fine residue are necessary.

In this way Cliver's technique measures the following materials used in making the mortar.

1. % Lime
2. % Sand
3. One of a) % Portland Cement
   b) % Natural Cement
   c) % Clay

The third technique studied is the test for cement content of hardened Portland cement concrete by the American Society for Testing and Materials. There is little doubt that this is a valid technique for the purpose for which it was designed, for the analysis of material known with certainty to be hardened Portland cement concrete. However, it often is used on historic mortars where there is no or little evidence for the use of Portland cement, and it is included here to examine its accuracy when used for this purpose.

This technique gravimetrically measures the amount of soluble silica present in a sample. Assuming that Portland cement contains 21% soluble silica the Portland cement content of the sample can then be calculated.

STANDARDS

To examine these three techniques, it would be best to use them to analyze a series of historic mortars of known compositions. Such mortars would have to meet the following restrictions:

1. The material must be homogeneous.

2. The nature and amounts of all materials used must be known accurately.

3. The standards must have never undergone leaching or contamination.

The nature of historic mortars is such that none of these criteria could be met; therefore, laboratory prepared samples were used as standards. These were made to duplicate realistically the Nineteenth and early Twentieth Century types found in Canada and they represent a broad range of compositions. For standards the following samples were prepared:

1. Lime mortar - by volume 1:3 lime:sand
   - by weight 8.40% lime
     91.60% sand

2. Lime mortar with calcareous sand - by volume 1:3 lime:calcareous sand
   - by weight 7.85% lime
     73.73% sand
     18.42% ground limestone

3. Lime mortar with clayey sand - by volume 1:3 lime:clayey sand
   - by weight 8.39% lime
     79.38% sand
     12.22% clay

4. Pozzalanic cement - by volume 2:1:5 lime:PFA:sand
   - by weight 9.53% lime
     4.32% PFA (Pozzalanic Fly Ash)
     86.15% sand

5. Portland Cement (hardened) - 100% Portland Cement

6. Portland cement with clayey sand - by volume 1:3 Portland cement:clayey sand
- by weight 20.37% Portland Cement
68.94% sand
10.69% clay

7. Portland cement mortar - by volume 1:3 Portland cement:sand
- by weight 22.26% Portland cement
77.74% sand

8. Roman cement mortar - by volume 1:3 cement:sand
- by weight 18.95% cement
81.05% sand

Information on all ingredients is given in the appendix. These samples were all thoroughly mixed and homogenized, water was added and the samples poured into molds. The curing of the samples was done to simulate as closely as possible the composition of historic mortars found in Canada. An important consideration was the extent of carbonation; experience in determining the calcium hydroxide and calcium carbonate levels by the thermogravimetric technique (Forrester, 1964) of historic lime mortars has shown that these are almost totally carbonated. Therefore standards based on lime (Nos. 1,2,3,4) were cured for 28 days in an atmosphere rich in carbon dioxide (10% CO_2). These were analyzed by the thermogravimetric technique and were almost completely carbonated.

The standards based on Portland or Roman cement, which experience has shown not to carbonate fully, were cured for 28 days at 100% relative humidity and 23°C (American Society for Testing and Materials Designation C511-68, 1971). Even without a carbon dioxide enriched atmosphere, thermogravimetric analysis showed these to have undergone carbonation of most of the free calcium hydroxide.

EXPERIMENTAL

The techniques used in all analyses were exactly as reported in the papers presenting the three methods.

RESULTS AND DISCUSSION

The technique of H. Jedrzejewska produced the results shown in Table I. Because the technique measures the amount of calcium carbonate, sand, and solubles in the samples after curing, the known values of the standards have been converted to these three values. This was done by determining the calcium carbonate and bound water contents of the samples by thermogravimetry and using the known amounts of other ingredients to calculate the sand and solubles concentrations.

There is a good correlation between the found and expected values for calcium carbonate, solubles, and sand, and the method meets its claims of being a simplified semi-quantitative technique. Furthermore, the soluble fraction is a qualitative indication of the potential presence of hydraulic components. Standards 1 and 2 contain no appreciable amount of material with potential hydraulic properties and have the lowest content of solubles. The remaining standards (3-8) all have components capable of being hydraulic. Thus, the technique could act as a rapid screening test to distinguish between those historic mortars which are non-hydraulic and those which may be hydraulic.

The technique of E.B. Cliver produced the results shown in Table II. The most interesting observation is that by using the colour of the fine residue as the sole criterion for qualitatively identifying the materials present, standards 3 and 4 are wrongly identified as containing Portland Cement and standard 8 is wrongly identified as being a lime mortar with a clay residue rather than a hydraulic mortar.

When a correct qualitative identification is made the quantitative results do not agree well with the expected results. Standard 1 has a relative error of 31% in the lime content which results from the incorrect assumption that the lime in the sample is only weakly carbonated. Standard 2 has a relative error of 238% resulting from the failure of the technique to recognize limestone as different from lime or carbonated lime. Standard 5 has a relative error of 91.9% in the Portland Cement content.

This last result indicates that hardened Portland cement may not contain 40% acid insoluble residue and as a result the theoretical basis for analyzing Portland cement is incorrect. However, analysis of standard 7 which contains only Portland cement and sand gives results which are at least semi-quantitatively correct for Portland cement; unfortunately it also shows the presence of an equal amount of lime in the mix which in fact was not there. Finally, standard 6 has a relative error of 93% in Portland cement content; this is caused by an error inherent in the technique, which includes the clay in the mix as Portland cement residue.

The technique of the American Society for Testing and Materials produced the results shown in Table III. As this technique is used to find the amount of Portland cement in an historic mortar only these values are listed. As expected, this technique performed well when analyzing standards containing no major sources of soluble silica other than Portland Cement; i.e., standards 1, 5 and 7. In all other cases it incorrectly showed the presence of Portland cement or showed an elevated Portland cement content (standard 6).

As stated before, this test was designed for hardened Portland cement concrete and in fact should not have been used on any of the standards except 5 and 7. However, when analyzing historic mortars it is rarely known with certainty that the necessary conditions for the accurate use of this technique are met by the sample and Table III illustrates the potential for error in the misapplication of this method.

CONCLUSION

Of the three techniques studied, that of H. Jedrzejewska seems to have the greatest applicability for the analysis of historic mortars. The characteristics it seeks to measure it does measure accurately, and one of these, the soluble fraction, can act as an indication for the presence of hydraulic components in the mortar.

The technique of E.B. Cliver did not succeed in correctly analyzing any of the prepared standard samples. It may be possible that this technique will give correct values for certain mortars; however, it would seem that in general the technique is not reliable.

The technique of the American Society for Testing and Materials accurately analyzed those standards for which it was designed; i.e., those having no significant source of soluble silica other than Portland cement. Given that this limiting condition is rarely known to be true with certainty in historic mortars, this technique would seem to have a limited applicability. Should the technique be misused, incorrect results could be obtained.

The normal question asked of an analyst working with historic mortars is "What were the materials and their proportions used to make the mortar?" While none of the three tests studied answers that question, the closest would seem to be the H. Jedrzejewska technique which determines the amount of sand and indicates whether or not the cement was possibly hydraulic.

If the question is to be answered, it seems unlikely that simple chemical techniques will be the solution. As the nature of mortars is largely dependant on their mineralogical composition the solution may lie in crystallographic and petrological techniques.

No matter which techniques for analyzing historic mortars are used, however, it is important that their reliability be assessed by analyzing known, realistic standards.

TABLE I. Analyses of Standards by the Technique of H. Jedrzejewska

		H. Jedrzejewska*	Known values and thermogravimetry results
1. Lime mortar	$CaCO_3$	10.1 ± 0.4%	10.0%
	Solubles	2.4 ± 0.7	0.8
	Sand	87.6 ± 0.3	89.2
2. Lime mortar with calcareous sand	$CaCO_3$	26.2 ± 1.3	25.9 **
	Solubles	3.0 ± 0.9	2.1
	Sand	70.9 ± 2.2	72.0
3. Lime mortar with clayey sand	$CaCO_3$	10.2 ± 0.2	10.4
	Solubles	13.2 ± 0.6	12.4
	Sand	76.6 ± 0.9	77.3
4. Pozzalanic cement	$CaCO_3$	11.0 ± 0.1	10.7
	Solubles	6.6 ± 0.4	5.55
	Sand	82.4 ± 0.3	83.8
5. Portland cement	$CaCO_3$	7.2 ± 0.4	7.0
	Solubles	92.1 ± 0.3	93.0
	Sand	0.6 ± 0.2	0
6. Portland cement mortar with clayey sand	$CaCO_3$	7.9 ± 0.1	8.5
	Solubles	29.6 ± 1.6	28.7
	Sand	62.6 ± 1.6	62.8
7. Portland cement mortar	$CaCO_3$	6.4 ± 0.4	5.8
	Solubles	27.4 ± 3.0	22.0
	Sand	66.2 ± 3.4	72.3
8. Roman cement mortar	$CaCO_3$	8.6 ± 0.6	8.6
	Solubles	16.1 ± 0.2	15.6
	Sand	75.3 ± 0.4	75.8

* Means and standard deviations of duplicates.

** Total calcium carbonate, including calcium carbonate from limestone; it is recognized the technique does not distinguish between carbonates from limestone and from lime.

TABLE II. Analyses of Standards by the Technique of E.B. Cliver

Standard	Colour of Residue		E.B. Cliver	Proportions used in the mix - Standard
1. Lime mortar	Tan	Lime	11.4 ± 0.0%	8.4%
		Clay	3.2 ± 1.2	0
		Sand	84.4 ± 1.4	91.6
2. Lime mortar with calca-reous sand	Brown	Lime	26.7 ± 0.1	7.9
		Limestone	0	17.9
		Clay	5.1 ± 2.2	0.7
		Sand	66.5 ± 2.4	73.7
3. Lime mortar with clayey sand	Grey	Lime		8.4
		P.C.	39.7 ± 0.5***	
		Clay		12.2
		Sand	71.8 ± 0	79.4
4. Pozzalanic cement	Grey	Lime	5.4 ± 5.8	9.5
		P.F.A.		4.3
		P.C.	12.5 ± 10.0	
		Sand	81.7 ± 4.5	86.1
5. Portland** cement	White	Lime	91.9 ± 2.4	0
		P.C.	8.1 ± 2.4	100
6. Portland cement mortar with clayey sand	Grey	Lime	2.0 ± 2.8	0
		P.C.	39.4 ± 3.5	20.4
		Clay		10.7
		Sand	58.0 ± 0.1	68.9

TABLE II. (cont'd)

Standard	Colour	of Residue	E.B. Cliver	Proportions used in the mix - Standard
7. Portland** cement mortar	Grey	Lime	16.6 ± 3.3	0
		P.C.	16.3 ± 5.7	22.3
		Sand	66.0 ± 2.5	77.8
8. Roman cement mortar	Tan	Lime	22.4 ± 1.0	
		Cement		18.9
		Clay	8.15 ± 0.1	
		Sand	67.4 ± 1.2	81.1

* Means and standard deviation of duplicates except as noted.
** Done in quadruplicate.
*** Sum greater than 100%.

TABLE III. Analyses of Standards by the Technique of the American Society for Testing and Materials

Standard	% Portland Cement used in the mix ASTM*	Standard
1. Lime mortar	0.0 ± 0.0	0.0
2. Lime mortar with calcareous aggregate	1.5 ± 0.5	0.0
3. Lime mortar with clayey sand	7.0 ± 1.4	0.0
4. Pozzalanic cement	3.1 ± 0.77	0
5. Portland cement	95.0 ± 1.3	100
6. Portland cement mortar with clayey sand	27.4 ± 0.2	20.37
7. Portland cement mortar	19.2 ± 0.1	22.3
8. Roman cement mortar	17.5 ± 0.4	0.0

* Means and standard deviation of duplicates.

APPENDIX: Ingredients used in mixes.

The following ingredients were used in preparing the standards.

Lime: Chemically hydrated lime, Joliette Brand, produced by Domtar Chemicals Group, Canada.

Portland Cement: Normal Portland Cement Type 10 produced by Canada Cement, Lafarge, Ltd.

Roman Cement: Produced by the authors by intimately mixing, calcining at 1100°C and grinding a mixture of 80% oolitic limestone and 20% clay. This is used to approximate the early hydraulic cements produced in Canada.

Pozzalanic Fly Ash: Alfesil, produced by C C Chemicals, Ontario.

Limestone: Fine grain limestone containing 96% calcium carbonate and 4% fine acid insoluble residue.

Clay: A grey ceramic grade clay.

Sand: Sakrete All Purpose Sand produced by the Flintkote Co. of Canada, Ltd. It has 1% passing a sieve with a mesh of 45um and a soluble silica content of 1.8 ± 0.3%

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ANCIENT MORTARS AS CRITERION IN ANALYSES OF OLD ARCHITECTURE

Hanna Jedrzejewska

SUMMARY

Ancient mortars are valuable evidence on old masonry techniques. There are two kinds of specialists interested in this evidence: technologists for technological reasons and archaeologists using mortars as additional criterion in chronological analyses of old masonry. Different purposes need also different analytical procedures, of specific analytic programs and accuracy. The relevant problems are discussed in the presented paper with special attention given to methodological aspects of the author's procedure of "comparative analysis", a system specially adapted to archaeological needs and based on a simplified program of determinations. The main objective of these determinations is to classify samples according to original recipes serving as a kind of "visiting card" for particular groups of masons.

Stressed is the urgent need for standarized procedures enabling to compare results coming from different laboratories. As yet the analytical programs and the measured parameters are individually chosen and cannot be compared.

Hanna Jedrzejewska, Chairman,
International Committee for Ethics
in Conservation (ICEC)

METHODOLOGICAL CONSIDERATIONS.

Generally, all kinds of materials used in ancient times by man to make things are evidence of past techniques and contain information that can be of value for different specialists. This also concerns mortars serving to keep together structural elements in masonry walls (stones, bricks) and usually considered as a source of information for technologists, not only from the purely historical point of view but also with the possibility of reconstructing old recipes and applying these in conservation procedures in mind. But recently also architects and archaeologists[+] started being interested in ancient mortars, though for a completely different reason. They are using mortars as a helpful criterion in analyses of chronology and interrelations of walls in old historical buildings and archaeological masonry relics.

Of course a different kind of information is needed in each case. The technologist wants to know everything about the mortar itself, its chemical composition, mineralogical and petrographic characteristics, mechanical and physical properties, etc. The archaeologist is not interested in mortar as such, but only in some characteristic properties of mortars enabling to compare them and classify into groups of similar origin.

The different kinds of sought information bear directly on the applied analytical procedures, because these have to be properly adjusted to the purpose of investigation. Some comments on this problem will be given in the paper. Considerations have to start with a review of basic factors connected with the mortar as such.

[+]For the sake of simplicity the term "archaeologist" will be used in the following text, on the understanding that an architect dealing with problems of ancient masonry in fact is also an archaeologist.

THE MORTAR

Masonry mortars are mixtures composed of a hardening medium plus an inert filler and eventually some additives improving the final properties of the product. For use, the mortar must have a workable plastic consistency. Then it dries out and hardens. The most common medium throughout the ages was lime (calcium hydroxide) obtained by slaking with water burnt lime produced from calcined limestone or other calcium carbonates. In the course of time calcium hydroxide combines with carbon dioxide from air, and is transformed again into calcium carbonate, acting as binder. This reaction is simple and common to all cases. Hence, lime mortars were for a long time of not much interest to technologists. (Taylor, 1957) states quite bluntly: "We may say then that the typical cement and mortar from the period before the 18th century was the familiar mixture of lime, sand and water". This is given as a conclusion of a brief survey of various kinds of mortars used in ancient times, and confirms the fact that modern technologist looks at mortars mostly from the perspective of Portland cement and "cement-like" materials.

Taylor's statement is a far going oversimplification. Lime mortars are far from being "simple" as concerns information and evidence.

First of all, different kinds of calcium carbonate materials could have been used for calcining, e.g. limestone rock, dolomitic rock, shell limestone, chalk, marble, etc. The slaking of burnt lime could have been done in open boxes (as it is the present custom), but also in sand covered heaps. In some cases, burnt lime (powdered or as half burnt limestones) was used directly without previous slaking. As yet, very little is known about these methods.

As next, comes the inert filler (though how much "inert" it is may be a matter for discussion), in most cases sand from the nearby river, from dry beds, or even from the sea shore. It could have been washed, calcined, sieved, etc. The granulation was very different, from very fine up to coarse, homogeneous and regular, or of mixed size, sometimes even with large pebbles included. The kind of inert filler and granulation was a characteristic feature of particular recipes. It was not only sand. Occasionally, crushed rock material was used and also crushed limestone (a very specific kind of limestone-lime mortars).

As concerns active components of mortars not enough is known about them. Used was crushed ceramic material, iron ore, charcoal. There are unconfirmed tales about eggs, blood, carcasses, etc. added to lime to make it stronger. Also clay and other siliceous materials could have been mixed with mortars to improve their "hydraulic" properties (though, it must be frankly said that our ancestors were rather unaware of this modern concept). Occasionally, fibrous materials were added for mechanical strength.

All this shows that old masons were quite ingenious in their recipes for mortars. Many of these mortars, even after hundreds of years, still have excellent properties, whereas our dear Portland cement is said to have a life expectation of less than a hundred years. But we still are not very successful at rèconstructing old recipes on the base of laboratory analyses. Maybe the analytical procedures are not satisfactory ?

Lime mortars are not the only kind of binding materials used in masonry. In certain rare cases gypsum was used. It still has excellent properties. Clay was mostly for structural elements (walls, raw bricks). Asfalt was found as mortar in ancient Mesopotamia, though probably only for some specific purpose.

Mortar like materials were used not only for joing elements in masonry but also for plasters and, occasionally, as artificial stone (Gothic sculptures, the walls of Alhambra, etc.). But this is a separate matter with a different set of independent recipes.

The basic requirement for good mortars was their reliability as concerns proper joining of structural elements in walls. And the mortar not only had to be good, but also to maintain the same properties in particular, separately prepared batches. This leads to recipes, effective, well tested and carefully followed. It also is logical to assume that each group of masons had its own, well guarded, professional secrets, and that there must have been some basic or smaller differences between the particular recipes (a kind of "visiting card" or "tracer").

ANALYTICAL CONSIDERATIONS

From the point of view of technology there is a lot of interesting information in ancient mortars. Some of these details connected with old recipes can easily be determined, and some only by very specific often undirect means. Used are conventional methods of modern technology. The chemical composition of a sample is, as a rule, expressed in percent proportions of oxides (Cao, MgO, Al_2O_3, Fe_2O_3, SiO_2, etc.). Carbon dioxide usually is determined by the loss of weight after calcining the sample at 900°C, seldom by volumetric methods. For petrographic and mineralogical characteristics samples are examined microscopically in thin polished sections. Mechanical and physical properties are determined by methods applied in modern building technology. More advanced investigations use chromatography, spectrography, X-ray analyses, thermography, electron microscopy, etc.

All these technological procedures are time taking and often need a very highly equipped laboratory. They need a large amount of mortar and are rather expensive. A multitude of experimental data is collected, but quite often all actual properties of samples, including the original ones as also secondary and accidental features, are examined together without proper differentiation.

To complicate matters some more, there are no standard programs of analytical procedures nor standarized methods of examination. Almost each laboratory has its own program of analysed elements and of analytical determinations. Even carbon dioxide is sometimes determined thermally and sometimes by volumetric means. This of course makes hardly possible a comparison of results obtained by particular laboratories and making some more general classifications. (Jedrzejewska, 1967).

From the point of view of archaeology analysing old masonry structures a completely different kind of information is needed and there also are other working requirements. The programs and procedures of investigation have to be adjusted to the intended purpose. The archaeologist is not interested in the mortar as such, but only in the particular characteristic features in original recipes still existing in the preserved material, and enabling him to classify his samples into groups of common origin. His reasoning is that when two samples of mortars show unmistakably, within limits of technological deviations, that they are practically "identical", it makes a strong argument for them as coming from the same time and being prepared by the same hands. But when supposedly identical walls show a definite difference in the used mortar this is an indication that they were erected on different occasions.

For this purpose simple, quick and inexpensive analytical procedures have to be applied to determine only these selected properties that have a direct connection with the original recipe. A large number of samples will have to be examined, eventually directly in the field. No high grade accuracy will be needed mostly because the mortars are rather inhomogeneous mixtures. But the analytical determinations have to be on a decent level of precision. Too many details have to be avoided.

The detailed technological programs are far from fulfilling these requirements. The characteristic features of original recipes are often lost in the mass of determined factors. Besides the methods are time taking, expensive and carried out with other purposes in mind. Occasionally, some of technological methods (mainly microscopic techniques) are used for a differentiation of particular groups of mortars. But for purely practical reasons the number of examined samples can not be large enough to satisfy the needs of the archaeologist. And some characteristics of the original recipe seem to be overlooked.

Taking all this into account, the author has started in 1950 to work with another analytical program, simplified as far as possible and specifically adjusted to archaeological needs. The procedure may be called "preliminary comparative analysis". It is simple, quick, inexpensive, can be carried out directly in the field, and is using small samples (very important when only small traces of mortar are available). Since then several thousands of mortars from different parts of the world and of very different date have been analysed and the results seem to be very effective, though the technologists are rather on the critical side of it.

COMPARATIVE ANALYSIS

The details of this method have been published elsewhere. (Jedrzejewska, 1967, 1960). Generally, the sequence of operations is as follows: well selected fragments of mortar (not in powder form) about 0,5g. each are crushed (separately), put in small crucibles and left for 24 hours to become air dry. At the same time all the visible characteristics of the mortar are noted (colour, texture, compactness, strength, pepples, fibres, lumps of lime, clay, etc.). The crucibles with mortars are weighed, enclosed in a gas volumeter (Jedrzejewska, 1967, 1960) and treated with HCI. The volume of evolved CO_2 is measured and recalculated to the corresponding weight of $CaCO_3$. Finally it is expressed in percents, in relation to the weight of the sample. This is value nr. 1. - the solution in the crucible is separated by decantation from the insolubles ("sand") remaining at the bottom. It is poured into a small glass tube (no water added !) together with all light suspensions and left for comparison with solutions from other samples. The remaining sand is washed by decantation and left to become air-dry. In case of need, both dryings, the preliminary and final can be made under a mild lamp. The dry sand is weighed and its weight expressed in percents of the sample weight. This is value nr. 2. The sand is preserved for observations and further comparison with sands from other samples.

The two values, nr. 1 and nr. 2 are added (it always is less than 100%) and subtracted from 100. This is value nr. 3. It represents "solubles", that means everything that is dissolved in HCI without evolution of CO_2, as well as all light suspensions floating in the solution.

These three values, helped with observations of mortar, sand, solution and, also, the rate of dissolution make a good base for classification and, as found from experience, are very sensitive to variations in recipes.

The relations between sand and lime are reflected in proportions between $CaCO_3$ and sand. It is not necessary to recalculate the $CaCO_3$ back to original lime putty (as is often tried in technological analyses), because the amount of $CaCO_3$ is just proportional to the original $Ca(OH)_2$, and for archaeological purpose that is enough information.

Depending on the kind of mortar and the time of its use the variations of proportions between (1), (2) and (3) may be very large, well above variations due to existing inhomogeneity of mortar. The content of $CaCO_3$, as expressed by (1) may be from about 1 - 2% (e.g. gypsum mortars) up to 95% or more (carbonate-lime mortars). The amount of sand may be from almost null to about 80-90%. Value (3) generally is between 4-8%. But in certain cases it may be 30%

or more (gypsum mortars, or with a high content of "soluble" silica). In some cases, the basic proportions of (1), (2) and (3) may be rather similar, but other secondary properties and the kind of used sand may become effective "tracers".

The positives in this method are:

- small amount of material for examination,
- all determinations carried out on the same sample (in technological analyses several samples must be taken from the mortar in order to complete the full analytical program, and this reduces the reliability of information).
- there is good practical information on the recipe as such and on the "hand" of the mason (thoroughness in maintaining the proportions, mixing, preparing the ingredients, etc.),
- there is no need to dry the sample, before analysis, to constant weight at 105°C which is a very cumbersome operation,
- the archaeologist gets information directly suited to his needs and does not have to sift the needed evidence from the large collection of technological details,
- the described procedure is inexpensive, quick and simple and quickly leads to sought information,
- analyses can be carried out even directly in the field. There is only need of a portable analytical balance (torsion type) and of a primitive arrangement for measuring the volume of CO_2,
- the procedure makes a good preliminary introduction for planning more elaborate and detailed research.

In fact, it is surprising how much information can be extracted from the mortar with this seemingly "primitive" technique, but only on the condition of very strict adherence to relevant methodological principles.

BASIC METHODOLOGY

This concerns principles that should be maintained in the course of investigation. They can be divided into three methodological steps:

(1) the collection of correct analytical data. Here belong matters of exactly defined purpose of investigation, of general analytical program, of necessary

analytical procedures, of representative samples, of critical evaluation of experimental findings (accuracy, reproducibility, precision, etc.) and of the way of presenting the results (classification, conclusions),

(2) the collection of information on the investigated object. This is necessary for having a properly established base for connecting information on mortars with problems of analysed architecture. The mortar in itself cannot speak. It can only answer questions posed by the investigated masonry. This has to be done in accordance with proper methodological principles (see "Instruction" at the end of this paper),

(3) the correct connection of analytical findings with the investigated object. Here come matters of keeping deductions and conclusions within allowed limits, determined as well by the investigated object as by information received on the mortar. No speculations or deductions extending over the factual material should be made. Critical restraint is necessary in the evaluation of interconnections between mortar and object. No "wishful thinking" should take place. It must be remembered that the mortar is not the sole criterion in these analyses but only a helpful addition to other arguments. Incidentally, it was found to be a very valuable source of information.

In the described method of "Comparative analysis" very careful attention is paid to methodological matters: as concerns analytical data. The purpose of investigation is to classify ancient mortars by differences in original recipes. Only relevant characteristic properties are determined (kind of ingredients, their proportions, some secondary features). The analytical program and the particular methods are adjusted to this purpose, and to the requirements of "simple, quick, inexpensive" in view of large numbers of samples having to be analysed.

Extremely important here is the matter of proper samples truly representative of what they are supposed to be. When the sample is not correct even the best analytical procedures will not help. The results will always be valueless. So, sampling has to be done very carefully by someone already well experienced. The joins have to be cleaned from extraneous matter and from parts of mortar that were exposed to external influences, and lumps of mortar have to be hammered out from the inside in amount sufficient for analyses and for storage as reference material (unconditionally imperative !). The place of sampling has to be carefully noted. In this way there is good chance that the mortar will be authentic and undisturbed by later influences.

Another basic element in analytical procedures are matters of accuracy, precision, possible errors, etc. The analytical determinations have to be precise enough for the intended purpose, but overaccurate determinations and too many details may obscure the sought relations.

It must be remembered here that there are two different factors bearing on the final precision of analytical results: the accuracy due to precision of analytical methods and the amount of accuracy depending on the properties of the analysed material. The final outcome of analytical determinations is a sum of both. And when the analysed material may be far from homogeneous (e.g. a mortar) the accuracy of results may vary from case to case. In the described "Comparative analysis" the accuracy of results was found well above the possible variations of mortar composition [1/] and the necessary reproducibility of results was found very satisfactory. As basic principle, two determinations were made for each mortar sample, and in case of large differences, two more parallel readings followed.

As concerns errors, the small size of analysed samples could bear on deviations in determinations but practice demonstrated that only in very heterogeneous mortars this became a problem. Another error was committed by not including Mg in the recalculation of CO_2 to $CaCO_3$. But there was, anyway, no difficulty in the classification of samples.

<u>As concerns information on the object</u>. The interest of the archaeologist in mortars lies in the possibility of using them as additional criterion in analyses of old masonry structures. So the archaeologist has to know at least something about the building before making a program for taking illustrative samples, and much more after getting the results of analyses. Samples also have to be taken by the archaeologist himself or under his guidance, in a number sufficient for a proper classification.

In making programs for sampling it must be remembered that ancient masons were very clever in their craft. For example, they used for the foundations of buildings mortars of the same basic characteristics but with proportionally more sand than for the upper walls. This could also happen in other cases, e.g. for floors, etc. Hence, an additional problem in sampling.

For proper effects of the comparative analysis of mortars also the analyst has to be well introduced into the kind of analysed object and its problems. Thus, he may be able to suggest some effective sampling procedure and he also will be more watchful for characteristic differences in mortars. But just bringing "a sample" to the laboratory for analysis makes the thing more mechanical with less chance for real success. And it happens quite often that samples are taken by "someone" from "somewhere" in the building, without a proper information on the object, and they are "somehow" analysed in the laboratory, without considering their true meaning and representative qualities. This leads to nowhere and the obtained results have no scientific value at all, even that they are used as scientific evidence. So, the analyst has to check, before starting his work, whether the received samples are properly connected with the building.

"Information on the object" means not only the knowledge of the ground-plan, form, style, destination, etc., but also information on its history, the supposed masons (monks of particular order, local groups, foreign masons, etc.) because this may be important when connected with mortars of specific characteristics, (see "Instruction").

<u>As concerns the connection of analytical findings with the investigated object</u>. This is the final step in the "Comparative analysis". It is a confrontation of the architect's hypotheses on the chronology and classification of walls in the investigated building with the analyst's classification of mortars into groups representing identity of recipes. The agreement between both sides may be good without further questions, but it may not be satisfactory, and then more thinking is necessary to find the cause of discrepancy. As experience shows it usually is the archaeologist that is wrong not the mortar. Anyway, in such cases additional sampling is necessary to check if the typical characteristics of mortars for the particular walls are properly determined.

The simplest application of the comparative procedure is for one single building. Step by step, very cautiously, this may be extended to complexes of buildings, and also to trace the activities of particular groups of masons [2/]. Interpretations here should be more tentative than decisive. Mortar should never be used as only criterion, but always in conjunction with other relevant information. Especially, no dating based on mortars is allowed. It can be tried only by comparison of mortar from the "unknown" with another mortar, from a well dated element. Plasters and other wall finishes can not be included into programs of comparison together with mortars used for masonry. They can only be compared between themselves, also by the same simplified analytical procedure.

FINAL COMMENTS

There is no doubt that ancient mortars are a source of valuable information for different specialists. This information must be collected in a proper way according to methodological principles and to the intended purpose. This already was discussed in some detail. A few general comments may be added:

(1) There is an obvious lack of standard procedures. It concerns as well purely technological investigations as all comparative procedures for the use of architects and archaeologists. There is a rather arbitrary selection of examined properties and of applied methods. Hence, there is no possibility of comparison of results and there are no methodological grounds for making some more general conclusions and comparisons,
(2) there seems to be a certain kind of competition between the conventional approach of technologists to the problem of mortars and the unconventional "simplified" procedures, even that the purpose of these two kinds of investigation is obviously different. The technologists do not like procedures that according to them are too "primitive", and the archaeologists complain that too many irrelevant (for them !) details are obscuring the sought relations,
(3) instead of this "competition" there might be a good cooperation. This in case when the preliminary classification and information on a large number of samples will be treated as a help in choosing proper representative specimens for further detailed examination. Also some observed

unexplained features in the first preliminary examination (e.g. soluble silica) could be nicely explained by technological analyses, etc.,

(4) in the described simplified comparative method it is necessary to have a good cooperation between the analyst and the archaeologist. And the analyst, before starting his examinations has to check carefully if the samples given to him were properly taken. This not always is observed and in consequence the analytical findings and classification may become pure nonsense without any fault in the analytical method as such,

(5) not only the lack of general standard procedures of examination makes impossible the drawing of a more general picture of mortar development and application, throughout the ages and in different geographic regions. There is some restriction in the possibility of open publication of results, because these are part of the archaeologist's research work and can be published only after the end of archaeological investigations. And together with all the other materials. On the other hand, when samples from masonry mortars are taken without cooperation with the archaeologist or architect they may not be representative of anything and thus have no scientific value. Unfortunately, this point is quite often overlooked and "samples" from different, just generally dated, buildings are taken as evidence, with no proper foundation of facts laid,

(6) it is very important to preserve for the future not only the results of laboratory research but also the original undisturbed mortars. This means preserving a "bank" of reference material, otherwise a collection of samples of known origin and a guaranteed "birth-certificate". Of course all analysed mortars should have their representants here. This is especially important and very urgent because during excavations and works of restoration, reconstruction, revalorization, etc. of ancient buildings old mortars are most often replaced with new cementing materials or infiltrated with consolidating agents and, thus, all important evidence is lost forever. This happens everyday. Something at least should be saved in the form of properly taken samples.

(7) a suspicion may be nursed that a large amount of presently available information on the technology and history of ancient mortars is not reliable and even valueless because of a large arbitrariness in research procedures. Conclusions based on these materials should be very critically reconsidered.

INSTRUCTIONS

These are basic directives for archaeologists and architects for proper sampling of mortars and for preparing standarized information on samples and on the investigated building.

I. Instruction for taking samples of ancient mortars for comparative analysis.

To obtain reliable results of analyses it is essential to use properly taken and representative samples:

(1) sampling has to be done by persons well acquainted with the building or its remains so as to ensure that a proper general program of sampling is prepared and the samples receive proper interpretation; it is desirable to include the analyst in the sampling operations,

(2) samples should be taken from the internal parts of the joins (except plasters), to avoid accidental changes due to weathering, infiltration or repair,

(3) the samples, as a rule, should be in form of lumps, not crumbled or powdered,

(4) the needed quantity for comparative analysis and for reference material usually is about 10-20 g., in one or a few compact fragments. There is no harm in having larger quantities,

(5) the exact spot from where the sample was taken must be carefully noted, eventually with the help of coordinates or a clear diagram. Never should samples from different spots be mixed together,

(6) to make certain that a particular kind of mortar is typical for a certain wall - minimum 3 samples should be taken from different parts of that wall, and analysed separately. If they prove to be identical within limits of practical deviations, their composition and properties may be considered as typical. Good practice makes this evaluation easier, and without this evaluation there is no ground for comparison with typical mortars from other walls,

(7) a single sample has no value for comparisons and can serve only as a source of some preliminary information. It has no scientific value and is not representative,

(8) in close conjunction with sampling it is necessary to collect as much information as possible, even in the form of hypotheses, on the investigated object and its problems. So that questions may be asked for the mortars to answer them. This information should be made in written form, to have clearly formulated statements. The simplest here is to use ready made forms to be filled in, one for each sample and one for the object. Seems tedious but is necessary from the methodological point of view and there is guarantee that nothing important will be forgotten.

Presented below are such forms as used in the author's laboratory. Filling them takes much less time and bother than when having to think each time afresh about items of information necessary for comparative analyses.

II. General information on the object.

This should be filled in as far as possible, eventually only with assumptions and hypotheses, of course stated as such:

1. Country:

2. Place:

3. Kind of investigated object: existing building (church, palace, fortification, etc.), or remains of a masonry structure, within existing building, or on archaeological site, evtl. sculpture or ornament in artificial stone, etc.,

4. Brief history of object, known or assumed, dating, general history of the place, owners or occupants, cultural contacts (religious, secular), due to planned or accidental influences, (marriages, wars, invasions, etc.),

5. Information about known ancient repairs, transfigurations, rebuilding and other changes concerning the basic structure of the building

or of its fragments,

6. Who the builders could have been: monks, local masons, masons of foreign origin. Was there a known monastic order, of what provenance, etc.

7. Purpose of investigation: comparison with other dated buildings, chronology of elements, assumed builders, differentiation between original and later added parts, preliminary information for a historian, technologist, etc. Program of planned investigation.

III. Information on the sample.

This has to be filled for each sample separately, as a kind of "birth certificate":

1. Nr. of sample: inventory, excavation identification, laboratory, etc. Depends on the particular adopted system,

2. Kind of sample: mortar, plaster, artificial "stone", moulding material, etc.,

3. Part of object from where sample was taken: foundation, external wall, internal wall, vault, flooring, basin, tomb, etc. Original or a later addition,

4. Exact localisation of sample: horizontal and vertical coordinates, with reference to standard zero or a well defined element of architecture. Depth in wall. Diagram of wall, with position of sample exactly marked,

5. Material of wall construction: stone (sandstone, limestone, etc.), raw or tooled, brick (size, colour), other materials, full wall or with rubble inside, etc.,

6. Environmental conditions of preservation of the investigated element: underground, in soil, clay, sand. In a grave or cellar. In dry or wet conditions (with comment). Above ground, exposed to dry or wet, cold or hot climate, windy, salty (sea) environment. Hidden within later structures, etc.,

7. General condition of the investigated wall: state of the wall, of its material, of the mortar joins. Adherence of mortar to material of

construction,

8. <u>State of sample</u>: strong, sound, weak, crumbly, very hard, partly crystallized, etc.

9. <u>Reason for taking the sample</u>: as dated standard, as representative example of building technique, for comparison with other samples, to establish chronology of walls, etc.,

10. <u>Probable dating of sample</u>: eventually only as hypothesis,

11. <u>Additional information</u>: relevant to the investigation but not included in the basic form,

12. <u>Name of person(s)</u> taking the sample and filling the record.
DATE.

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The presented orderly and systematic arrangement of standard information is not only directly useful for comparative analysis but makes also a good starting point for further historical deductions and for detailed technological investigations.

NOTES AND REFERENCES

TAYLOR, F. S. A History of Industrial Chemistry. London, 1957, 55-58.

JEDRZEJEWSKA, H. Investigation of Ancient Mortars, in "Archaeological Chemistry", University of Pennsylvania Press, Philadelphia, 1967, 147-166. Extensive bibliography and a critical review of procedures is accompanying the description of the author's method of simplified analysis.

JEDRZEJEWSKA, H. Old Mortars in Poland: A new method of investigation. Studies in Conservation, 1960, Vol. 5, No. 4, 132-138.

1/ To check the accuracy of determinations of CO_2 and sand, as proposed for the comparative analysis, the following tests were carried out:

<u>A</u>. CO_2 was volumetrically determined in four samples of chemically pure $CaCO_3$, each about 0.25 g., and the amounts compared with theoretical yield. The accuracy of determinations was within $\pm$ 0.5 ml. of CO_2, corresponding to $\pm$ 0.0022 g.$CaCO_3$. This accuracy was considered as excellent for the intended kinds of analysis;

<u>B</u>. for sand two kins of tests were done. In the first series a larger fragment of mortar was powdered, divided into four parts according to technological standards, and sand was determined for each part separately. In the second series, four <u>separate</u> samples were taken from the same larger fragment of mortar, separately powdered and analysed. In the first series the results of sand content determination were fairly consistent (between 75,3 and 76,9%, with average of 75,6%). In the second series the results were: 66,0-76, 3-77, 8-81, 5%, with average 75,4%. It was decided that for very heterogeneous mortars not two but four determinations gave better results, but it also was decided not to operate with averages because in this way one more characteristic feature of the mortar, the inhomogeneity, was lost.

2/ There are some specific kinds of mortars that were used only at a certain time and in certain places, and then, have completely disappeared. Here belong gypsum mortars, carbonate-lime mortars, as well as mortars with fibers, with crushed ceramic material, etc. Some specific recipes seem also to be connected with Cistercian masonry, with Benedictines, etc. The Greeks had some excellent recipes (still remaining a technological mystery), the Romans used grainy sands with pebbles for their "ciments", etc. In Gothic times a much higher proportion of sand was used than before and after. All this information comes from the preliminary comparative investigation, and can be of use not only to archaeologists but also to historians.

As example of all this two publications may be mentioned:

DE BOUARD, M. Manuel d'Archéologie Médiévale, Societé d'Edition D'Enseignement Supérieur, Paris, 1975, 302-307.

In the section on analyses of masonry mortars discussed are methods of analyses, partly based on J.Jedrzejewska's simplified determinations and partly on the own methods of the research laboratory at Caen (France).

MALESANI, P., MANGANELLI, C. and VANUCCI S. Applications of mineralogical-petrographic techniques to the study of S. Reparata Basilica (The Old Cathedral of Florence). In "The Conservation of Stone", I, Bólogna, 1975, 81-88.

Investigations of mortars and bricks enabled correlations between these materials from dated elements and the unknown, otherwise non datable, ones. The hypothesis of characteristic recipes for each phase of building found very good confirmation. About 80 mortars were analysed. No details are given about analytical details or about the determined parameters.

Both publications confirm the value of ancient mortars as source of important information. Discussed are many practical and methodological points, but there is nothing about standarized procedures to be followed in all cases of mortar investigation. The same happens with many other publications and this makes any more conclusive generalizations impossible.

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L'ANALYSE DES MORTIERS ANTIQUES; PROBLEMES ET RESULTATS

FRIZOT Michel (1)

RESUME

A review of the problems of chemical analysis in the field of studies of ancient mortars (especially roman provincial mortars). The author gives an account of methods used , and results obtained in the articles he gathered, as from his own experiments. The main constituants of mortars are reviewed: aggregate, lime and the ratios of mixing. The study of mortars is useful to archaeologists, for the knowledge of monuments excavated, and of the ancient techniques. But it can be helpful, and seems necessary for the restoration and preservation.

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INTRODUCTION

Les mortiers de l'Antiquité gréco-romaine ont fait l'objet d'assez nombreuses études chimiques ou physico-chimiques (nous en avons répertorié plus de trente qui apportent des résultats nouveaux et précis); mais elles sont le plus souvent très ponctuelles , ne concernant que quelques échantillons très localisés, et ne répondent généralement qu'à un souci de curiosité qui n'englobe pas de considérations générales sur le matériau antique. Il ne saurait être cependant question de négliger les renseignements de ces études , ne serait-ce que pour les résultats acquis, mais aussi en raison des diverses manières d'aborder les mortiers et de les "interroger" par des moyens scientifiques. Nous avons recensé ces études, en y ajoutant nos propres résultats de recherche qui portent sur environ 160 échantillons pour des analyses assez complètes, et nous faisons ici le point des problèmes que posent ces analyses et de leur apport à la connaissance de la technique antique, technique dont on doit évidemment tenir compte dans toutes les questions de restauration ou consolidation. Il semble que trop souvent des restaurations soient entreprises sans une bonne connaissance du matériau traité, aussi bien pour sa composition que pour son mode de réaction aux agents extérieurs ou aux produits de consolidation.

L'ANALYSE CLASSIQUE

Nous avons considéré le mortier d'un point de vue général, aussi bien dans sa fonction de mortier de liaison que de matériau constitutif des couches d'enduits; les questions d'ordre technique y sont souvent les mêmes. Les mortiers doivent être à priori regardés comme un mélange de chaux et d'agrégat (chaux éteinte qui se carbonate avec le temps). Quels sont tout d'abord les moyens d'analyse des mortiers et , partant, le type de question qui est posée par l'analyse ? Les articles sur ce sujet étant pour la plupart anciens, l'analyse est "classique" c'est à dire consiste en dosages d'insolubles et en solubilisation / insolubilisation de sels. Pour les mortiers, la règle est l'attaque à l'acide chlorhydrique dilué (environ 1/5) qui solubilise le carbonate de calcium en libérant le gaz carbonique (1). La réaction est utilisée pour le dosage de la chaux contenue dans le mortier (en état carbonaté) , soit par pesée de l'insoluble et calcul du pourcentage de carbonate par différence, soit par mesure de la quantité de gaz carbonique dégagé, qui renvoie au carbonate de calcium. Il ne s'agit là que d'approximations, la première méthode parce qu'elle suppose que seul le calcium est solubilisé, alos que d'autres éléments le sont (aluminium, fer, silice), la seconde parce qu'elle ne rend compte que du calcium réellement carbonaté. La complexité du mortier en tant que mélange d'agrégats variés sur le plan chimique, et de chaux , ne facilite pas ce travail de lecture des résultats et ne permet pas d'atteindre à une certitude absolue quand à leur interprétation. Il faut en effet compter avec l'évolution du mortier dans le temps (désagrégation, interaction d'éléments) et à l'action de l'acide sur l'agrégat vis à vis duquel il ne se comporte pas forcément de manière neutre. On le voit en particulier à propos d'une mesure qui est faite par de nombreux auteurs: celle de la silice soluble , qui est censée venir soit de la chaux hydraulique , soit de l'agrégat si celui-ci est du type pouzzolane. Or, il semble que, dans les conditions de l'analyse, l'acide dilué puisse attaquer partiellement des agrégats qui ont évolué chimiquement depuis 2000 ans, ou attaquer le tuileau, ou les pouzzolanes, qui sont très sensibles à l'acide ; l'acide servant à la mesure ajouterait ainsi , sans qu'il soit possible de le contrôler, un certain pourcentage de silice. D'où le doute que l'on peut avoir à l'endroit de cette mesure lorsqu'elle est prise pour preuve, par exemple, d'une chaux hydraulique (2).

Par les mêmes méthodes classiques (précipitation par l'ammoniaque), on dose souvent les oxydes de fer et d'aluminium. Mais là aussi, selon la nature de l'agrégat en présence de l'acide , on ne peut être certain que ces oxydes proviennent entièrement de la chaux.. Le tuileau, les granits, les calcaires impurs , les argiles de l'agrégat sont attaqués également et produisent de ces oxydes pour peu que l'action acide soit un peu prolongée.

L'analyse classique d'un mortier dose donc, mais d'un pointde vue quelque peu utopique les éléments constitutifs suivants: chaux carbonatée, oxydes de fer et d'aluminium, silice soluble, agrégat insoluble. Pour notre part, nous avons préféré une méthode de dosage globale, qui intègre les composants de l'agrégat à ces résultats et considère donc le mortier comme un tout non scindable. Nous avons pratiqué pour cela (en ajoutant la magnésie à la table des dosages) la spectrométrie d'absorption atomique, puis, plus récemment, la fluorescence X . Ces mesures permettent de comparer pleinement entre eux les mortiers et d'établir ainsi des typologies, ce qui était l'un des buts de notre travail (3), qui n'aurait pu aboutir par les méthodes classiques, trop sujettes à variation. Nous avons ainsi obtenu des compositions globales de l'échantillon en $CaCO_3$, SiO_2, Al_2O_3, Fe_2O_3, MgO, et nous avons comparé les échantillons en reportant les résultats sur un diagramme triangulaire SiO_2 / $CaCO_3$ / somme des oxydes métalliques. Nous n'ignorons pas le côté artificiel de telles mesures (cependant doublées par des calcimétries et d'autres mesures plus "parlantes") mais elles permettent au moins des comparaisons très utiles qui trouvent leur apllication au niveau de la fouille archéologique.

L'AGREGAT

L'agrégat est ; à l'oeil nu, le constituant le plus évident d'un mortier. On s'intéressera, en premier lieu, à sa nature immédiatement sensible, que l'on peut qualifier de géologique. Or, si nous résumons les observations faites dans le monde romain, il apparaît que cet agrégat est extrêment varié, et c'est même son caractère premier. Loin de constater, comme le laisserait penser Vitruve, que nous avons un choix très précis, il semble assez souvent que l'on a utilisé le matériau dont on disposait au plus près, y compris la terre argileuse. Le sable siliceux est bien sûr l'agrégat de prédilection du mortier antique, mais sous toutes ses formes locales: sable de rivière ou carrière, ou sable de mer. La pouzzolane est utilisée dans certaines régions d'Italie mais l'importance de sa présence reste à préciser pour ce pays. On rencontre souvent aussi un agrégat calcaire, lorsque le substrat local est dépourvu de formations siliceuses. Enfin , l'Antiquité fait un large usage du tuileau (tuiles et briques broyées) et ses vertus peuvent être comparées à celles de la pouzzolane; on trouve ainsi le tuileau en majorité lié aux lieux humides ou en contact direct avec l'eau. Mais les mélanges sont extrêmement courants: siliceux + tuileau, calcaire + tuileau , etc...

L'argile est aussi, à titre d'impureté ou d'élément introduit sciemment , un constituant présent dans tous les mortiers. Et ce matériau est souvent sous-estimé par les auteurs, qui , bien naturellement, se sont d'abord intéressé à la matière la plus noble et la plus résistante, avec le secret espoir, parfois, d'en percer quelque mystère, celui du mortier romain. Or, ce mortier n'a pas toujours été aussi bon que le laissent supposer les vestiges qui nous restent (et qui se sont perpétués précisément par l'excellence de leur matériau) et l'argile entre pour une bonne part nous semble -t-il , soit avec de la chaux, soit seule, dans la fabrication des mortiers antiques; à ceci près que ce matériau est beaucoup moins souvent conservé en bon état que son homologue plus renommé. Nous rejoignons ici un autre aspect de l'archéologie romaine, la mise à jour précise de l'utilisation de la terre dans la construction, et, entre autres , en tant qu'élément de liaison ou d'enduit (4).

La connaissance de l'agrégat des mortiers antiques est très souvent complétée par le tracé des courbes granulométriques cumulatives; elles ont l'intérêt de permettre des rapprochements, des comparaisons précises, et de créer des groupements typologiques par une transcription visuelle de la répartition des tailles de grains de sable. Mais la courbe granulométrique ne servirait qu'à des comparaisons visuelles si elle n'était assortie des indices granulométriques qui, numériques, sont exploitables avec la même facilité que d'autres mesures physiques. Peu d'auteurs les emploient , mais il paraît cependant indispensable de déterminer par exemple la médiane d'un sable; elle se situe d'après nos résultats et les études antérieures, avec un maximum autour de 1 mm et il est clair aussi qu'elle est plus faible pour des mortiers d'enduits que pour des mortiers de liaison de mur. Pour l'un des sites que nous avons étudiés , en Bourgogne, le sable de rivière utilisé était particulièrement fin (médiane à environ 0, 25 mm). Le "sorting index" (indice de triage) nous renseigne aussi sur une autre caractéristique de l'agrégat: son triage, c'est à dire la régularité de répartition symétrique des mailles de grains autour de la médiane; il sera beaucoup plus faible pour un sable d'alluvion, bien trié, que pour un agrégat constitué de matériaux tout-venant. Les courbes et indices granulométriques sont autant de moyens de caractériser un mortier, qui n'auraient pas grand intérêt s'ils étaient isolés mais prennent tout leur sens par les rapporchements, les regroupements que l'on peut effectuer, à l'échelle d'une maison, d'un site, d'une région, ou d'une époque.

LA CHAUX

L'autre constituant important du mortier est la chaux. On ne peut évaluer son pourcentage exact que dans le cas d'un mortier de sable siliceux, qui ne contient donc pas lui- même de carbonate, ou d'un tuileau. Il conviendra de vérifier avant une mesure que nous sommes bien dans ce cas, faute de quoi la quantité de chaux serait surestimée, et ce type d'erreur est maniteste dans plusieurs études anciennes. La teneur en chaux carbonatée, d'après nos mesures et celles que nous avons répertoriées, se situe entre 5 et 35 % en poids, avec le maximum des échantillons entre 20 et 35 % . La nature de la chaux qui a donné ce carbonate de calcium est souvent sujette à caution et très discutée; la question la plus importante du point de vue de la technique antique est de savoir si nous avons une chaux aériennne grasse ou une chaux hydraulique. La différenciation, peu sûre, se fonde sur la présence de silice soluble dans le mortier, et on peut renouveler ici les critiques faites précédemment concernant la mesure de ce constituant.

La meilleure réponse à apporter est l'analyse de chaux romaine d'origine, que l'on retrouve parfois dans les fouilles. De même, les couches supérieures des enduits, constituées le plus souvent de chaux et de marbre broyé n'introduisent pas de risque d'erreur dû à l'agrégat et présentent une chaux dans sa constitution d'origine. A l'analyse, on ne trouve dans ce cas que des chaux grasses ou très faiblement hydrauliques comme nous avons pu la vérifier nous même. Nous avons d'autre part étudié de ce point de vue quelques calcaires provenant de chantiers archéologiques, et susceptibles d'avoir été grillés pour obtenir de la chaux dans l'Antiquité; ils donnent le même résultat. Il ne semble donc pas que les Romains aient eu conscience de l'hydraulicité de la chaux obtenue par la calcination de calcaires marneux et il est probable que, comme pour les autres matériaux , les conditions fortuites locales aient imposé tel calcaire qui fournit une chaux hydraulique (ce pourrait être le cas dans le massif de l'Eifel, étude de Grün). De plus, il faut différencier, dans l'appellation, ce type de mortier "hydraulique" (si on parvient à le mettre en évidence) d'un autre type obtenu par mélange d'une chaux grasse et d'un agrégat "hydraulique" comme la pouzzolane. Ce mot "hydraulique" est d'ailleurs d'une utilisation quelque peu ambigüe et trop différenciée pour être manié sans danger lorsqu'il s'agit de l'époque antique. Il nous faut nous garder de juger la technologie de cette période avec le regard que nous a légué l'industrie du 19 ème siècle.

LES PROPORTIONS

Connaissant les poids respectifs du carbonate de calcium et de l'agrégat dans un mortier, on peut évaluer les proportions du mélange initial (proportions en volume) comprenant l'agrégat et la chaux éteinte (donc non carbonatée). Il ne peut s'agir que d'une évaluation, car on ne connaît pas la quantité d'eau qui entre dans la fabrication de la pâte de chaux, mais on peut admettre comme principe qu'elle est de 50% d'eau. Les proportions que l'on a évaluées sont alors très variables, mais avec un maximum entre 1/2 et 1/3 (chaux à 50% d'eau / agrégat,en volume), ce qui correspond aux principes de Vitruve (1/2 ou 1.3 selon que l'on a du sable de rivière ou de carrière). Il ne faut cependant pas cacher que ces limites sont souvent dépassées inférieurement ou supérieurement. L'énoncé de Vitruve apparaît alors comme une solution moyenne de simple bon sens et de pondération, qui n'implique pas forcément la connaissance, par l'artisan, des recettes figurant dans un traité d'élite, ni même une recette colportée par la tradition professionnelle.

LES DIFFICULTES D'INTERPRETATION

On le voit, le jugement que l'on peut porter sur un mortier antique , c'est à dire la relation des résultats trouvés et de ce que nous croyons pouvoir attendre à travers les connaissances établies par le savoir archéologique, n'est pas chose facile, tant il s'avère aventureux d'accorder à un artisan antique une conscience précise de ses gestes, alors que le hasard ou le bon sens artisanal ont pu souvent le guider.C'est par des moyens statistiques seulement que l'on peut espérer mettre en concordance de telle données encore fragmentaires. Dans le domaine des enduits, par exemple, il est bien rare que l'on découvre une technique répondant aux prescriptions de Vitruve qui requiert sept couches superposées, à tailles de grains d'agrégat variables. Mais on a cependant un archétype d'époque romaine, assez constant dans toutes les provinces, à une ou deux couches de mortier de chaux et de sable et une couche préparatoire à majorité de chaux, avec ou sans marbre broyé.

Nous avons noté, comme point déterminant des mortiers antiques, la variété de l'agrégat. Elle se vérifie à plusieurs niveaux, et en particulier concernant la destination d'emploi du mortier. Pour les enduits recevant des peintures, on préfère un agrégat siliceux, non argileux , bien lavé, et plus fin que pour un mortier de liaison. Le tuileau est présent, seul ou en mélange, dès qu'il s'agit d'obtenir une étanchéité (jointoiement , enduits de bassin,aqueducs, emplacements humides) et on le rencontre dans ce cas, dans l'une ou l'autre, ou dans la totalité des couches d'enduits.

La technique dépend en fait souvent de la géologie locale ; il ne semble pas que, pour une construction normale, on ait fait des importations lointaines. On ira jusqu'à utiliser la terre locale, sans chaux, en terrain granitique, ou le sable marin, avec ses coquilles, en bordure de mer, et bien, naturellement , on se contentera pour l'agrégat, du substrat sur lequel s'édifie la construction. Ces considérations n'excluent pas les connaissances techniques des Anciens, qui sont manifestes dans les fouilles, mais il est encore difficile de les cerner et d'en comprendre la véritable nature, peut-être plus proche d'une superstition ou d'un occultisme que d'une connaissance cartésienne raisonnée.

PERSPECTIVES

L'étude des comportements des mortiers dans le temps présente un grand intérêt du point de vue de la restauration , en particulier si l'on pose la question de cette façon: serait-il juste et rentable de refaire des mortiers "à l'antique", comment évolueront-ils dans le futur, ou faut -il les remplacer par des produits modernes, ou faut-il seulement les compléter, les consolider par des imprégnations?

Ce sont là des questions courantes dans le domaine des matériaux de construction , mais il est difficile d'y répondre pour le cas des mortiers. La technologie moderne a en effet laissé dans l'ombre l'étude de ce matériau simple depuis qu'il a été remplacé par le ciment au 19 ème siècle. Ainsi, nous ne connaissons pas précisément les phénomènes de la prise, ni la cinétique, ni la chimie de cette transformation physique. Ainsi, nous voyons mal quel est le rôle de la carbonatation et sa vitesse, quel est le rôle de la cristallisation de la chaux éteinte, quelle est l'importance de la phase colloïdale (5).

En ce qui concerne l'évolution du mortier dans le temps, et cette "bonification " qui semble être le fait du mortier romain (de certains mortiers romains), nous ne connaissons pas l'éventuelle formation de silicates par la réaction chaux- agrégat (qui a lieu avec les pouzzolanes, avec le tuileau, et aussi sans doute, mais à quelle échelle, avec d'autres agrégats). En milieu humide, ceux -ci ne se comportent certainement pas de façon neutre. Nous ne connaissons pas davantage le mode d'action du tuileau avec la chaux (porosité, dissociation des argiles à la cuisson). Et ces questions importantes ne nous paraissent pas avoir été traitées scientifiquement . Enfin, nous n'évoquons que pour mémoire les propriétés physiques telles que la porosité du mortier, ou sa résistance, qui sont évidemment liées à sa composition chimique et géologique.

Les résultats dont nous avons connaissance à l'heure actuelle nous paraissent avoir une double utilité: d'une part aider à la connaissance des sites archéologiques, des campagnes de construction, des variations chronologiques; d'autre part éclairer d'un jour nouveau la technique antique et l'interpréter avec un regard critique en fonction des données locales.. Mais il est une troisième voie qui peut trouver profit à l'analyse des mortiers antiques, celle de la conservation et de la restauration. La connaissance précise du matériau doit y intervenir comme préliminaire à la définition d'un mode d'intervention lorsque l'on envisage de conserver une part importante du monument en place, avec son matériau d'origine. Nous avons voulu montrer que, si les questions posées sont assez bien définies, il manque encore beaucoup d'études sur ce sujet pour que les diagnostics y puissent être clairs.

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ANCIENT MORTARS AND CONCRETES - DURABILITY ASPECTS

Roman Malinowski

SUMMARY

Ancient lime mortars and concretes were investigated in situ and tested in laboratory. Materials used and technology applied were analyzed. The use of the protection technique of walls and floors by means of polishing the multilayer mortars was confirmed. Highly durable concretes were obtained by using different binders and aggregates. Entruded air was found in concrete exposed to frost. Oil-quicklime mixture for jointing pipes in pressure conduits and a method of protection of pavements by means of oil impregnation (given by Vitruvius) were described. The scientifical character of the empirical solutions was discussed.

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INTRODUCTION

Mortars and concretes of unusual durability have been discovered in remains of many remarkable ancient building and engineering structures (Davey, 1961). Despite of being in use during centuries and exposed to severe environment these mortars and concretes often show a much better permanence than natural stones or burned bricks of the same structure, and sometimes better durability than modern concrete exposed to similar conditions (Malinowski et al, 1961).

This publication is compiling the author´s previously presented investigations with results of new tests and literature data (Malinowski, 1979[1] and 1979[2], Idorn, 1959). Examples of structures and material with excellent durability are given. Studies in situ were complemented with laboratory tests in which physical and mechanical properties, the microstructure and chemical composition of the material were analyzed. Based on this investigation an explanation in modern terms of the durability of the material and of the methods applied in construction is presented and unsufficiently clarified problems pointed out. The materials were studied in following chapters:

- lime mortars for protection of structural components
- concrete in structures
- special material solutions

DURABLE LIME MORTARS

The protection of a weaker substratum of walls, columns and floors by careful polishing of fresh lime mortar has its origin in the early antiquity. The first known use of this procedure is to relate to the Mask of Jericho (7000 B.C.). Many examples of high quality polished mortar were confirmed in Mycenaean and Minoan settlements (1500 B.C.), in the famous cistern of Mycenae and in many buildings of Phaestos and Malia in Crete etc. The Greeks took over the antique technique and later on the Romans applied it in hydralic structures. Floors, walls and columns in cisterns and lining of aqueducts were covered by a unilayer or multilayer mortar finely polished. Examples of the use of unilayer mortar are the Greek water cistern of Megara (500 B.C.) and the Roman aqueducts of Pont du Garde (100 A.C.). The multilayer one is to be found in the King Salomon Cisterns, the Herodian aqueduct of Caesarea (0-100 A.C.) and others.

Lightweight lava aggregate was often used in mortars - in the buildings of Forum Romanum in Ostia, Pompeii, Herculanum this mortar is to be found. The very good permanence of such a polished mortar is to explain by the low water absorption and desorbtion of the aggregate which, acting similarly as entruded air, thus are affecting the swelling and shrinkage of mortar.

Vitruvius describes the polishing of mortars as an old Greek procedure. He stated that careful polishing helps to restrain shrinkage and cracking. An explanation of the mechanism of polishing and of the influence of the procedure on the hardening, structure, properties and durability is given. The polishing, being a grinding of the lime, carbonate or pozzolana of the mortar creating a dense capillary system at the surface, is simultaneously accelerating the carbonation and (or) the hydration process. The finely polished, hardened surface is not only restraining shrinkage and cracking, but also checking the creation of lime sediments on the walls of cisterns and aqueducts due to a better flow of water. The removal of such sediments created is also eased. The function of the different layers of the three- and sixlayer sandwich mortar was also studied. The lightweight layer, consisting of cool ashes, is a bond layer, the carbonated one is a shrinkage-preventing layer and the polished pozzolanic one (of grinded ceramic) is assuring impermeability and strength (figure 1).

DURABLE STRUCTURAL CONCRETE

Concrete - "opus caementitium" - is commonly supposed to be a Roman procedure. The use of mortar and plaster in Greek buildings, mentioned and in detail described by Vitruvius, indicates that the concrete probably was known in the pre-Roman periods. Studies of the cistern of Kameiros - Rhodos (500 B.C.), where pozzolanic concrete is covering the walls of the pool, are confirming this statement.

For structures in which high strength of concrete and high durability was needed, in protecting walls, harbours, in aqueducts and buildings constructed in severe cold climate, lime-pozzolana binder was used generally. In harbours of Ostia, Puteoli and Villa Polia (near Sorrento) and in many buildings of the Capitolium the use of this concrete was confirmed. Of special interest are the very good preserved remains of the many hundred meters long, crackless town wall of Ampurias, Spain (0-50 A.C.), made of lime-pozzolana concrete and built without any dilatation (figure 2).

The opinion that such a binder was used in more important structures exclusively was not confirmed. In many Roman hydraulic and marine structures and in mineral springs, in which high durability was required, limebased concrete was often used. As examples are to be mentioned the concretes of the North-German aqueducts in Eifel and others. In both lime- and pozzolana-lime concretes different types of coarse aggregates were used. Natural gravel of different origin, crushed aggregates of heavy basalt of porphyry, granite or limestone, artificially crushed aggregates of clay bricks and tiles and light lava were used. In one case gape grade aggregate concrete was found. Vitruvius describes in detail sand of different quality and recommends its application for different functions. The lime:sand-proportion was adjusted to the sand quality (figure 3).

To improve workability and first of all the durability of concrete, air entrainment was added in concrete exposed to frost action. That was confirmed by the very interesting microscopical investigation by Idorn. Lightweight concrete made of lava-aggregate was often used in Roman buildings to get better insulation and to reduce the weight. An example is the top part of the dome in Pantheon (figure 4).

The application of the rules for selection, proportioning, mixing and compaction of concrete, recommended by Vitruvius, were confirmed in concretes of many structures located in different parts of the former Roman Empire. The examples of crackless structure of many Roman buildings indicate a proper choice and adjustment of the material and the applied technology and good understanding of the behaviour of the material, applied to different types of structures and the environmental conditions. As examples can be mentioned thick concrete walls with slopes, the domes and arches casted of massive concrete (as in Pantheon, Basilica of Constantin - Maxentius, and others). The curvature of the structure is resulting in uniaxial vertical creep deformation of concrete and restrained creep in the horizontal direction, thus compensating the shrinkage deformation and avoiding cracking (figure 5).

SPECIAL SOLUTIONS

In the work are presented some unusual and interesting ancient material solutions. Gypsum concrete used as binder in the Pyramides was found also in the walls of the Mycenaean Palace (1500 B.C.) and as material connecting giant blocks in the ancient harbour of Kition - Cyprus (600 B.C.?). This uncommon case of the durability of gypsum concrete in marine structures is worth studying (figure 6).

Another interesting engineering solution is the expanding paste for fitting joints of pipes in pressure aqueducts. The paste found first in Knossos was confirmed in many Greek and Roman ducts made of lead, clay and stone. The joints assured that the aqueducts could resist a high pressure of many atmospheres. In the famous aqueduct made of lead pipes, leading water from the castle of Pergamon to the town, the pressure was nearly 20 atmosphere. The sealing material used was, as described by Vitruvius, of quicklime mixed with oil and probably also with finely grinded limestone (as in Vitruvian stucco mortar). This was confirmed in simulation tests performed by the author (figure 7). A similar paste was found also in mosaic floors of Roman baths, where the function of sealing and waterrepellency was somewhat like. Another material for sealing joints in ancient ducts due to expansion, montmorillonite, was found in one of the aqueducts of Pergamon.

Iron bars to reinforce stone beams were found in Propylee. Nothing is known, however, about the applied protection against corrosion of the bars. To assure durability of floors exposed to frost action was recommended by Vitruvius to impregnate the mortar jointing of the stone plates by means of oil. A similar method to improve the durability of modern pavements is commonly used in the United States.

GENERAL REMARKS

The ancient unilayered and multilayered mortar, polishing techniques for the protection of the substrate being of weaker material, the different types of concrete based on different binders and aggregates, the use of air entraining admixture to improve durability, the impregnation with oil of the jointing mortar, the ancient expanding sealants, and other solutions are impressive examples of successful solutions of ancient engineering. Studies of these techniques reveal that the ancient engineers had a good understanding of factors involved in creation of engineering structures, their protection and maintenance.

The lack of modern scientific methods and detailed specialization were compensated by an experience based on tradition and knowledge of a more general character. In our own times invention and engineering solutions often proceed a proper scientific explanation. The ancient engineering practices were used for centuries without a clear understanding of the scientific basis of the techniques used. Nevertheless, they were eminently successful. Many of these techniques are of interest and importance to modern concrete engineers, historians of science and technology and archaeologists.

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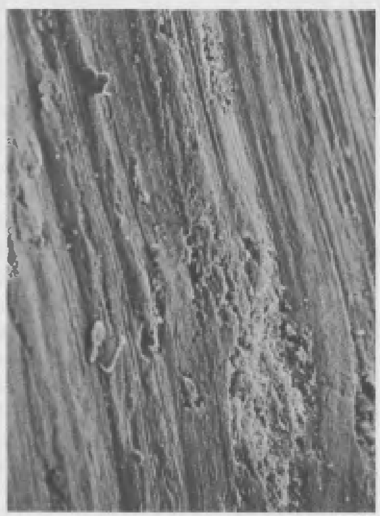
C



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D

Figure 1. Polishing technique of lime mortar.

A. The Mask of Jerico, 7000 B.C. (Kenyon, 1970)
B. Painted wall in Phaestos - Crete, 1500 B.C.
C. Multilayer mortar in the aqueduct lining of Caesarea, 0-50 A.C.
D. SEM micrograph of a polished surface of mortar. Detail of C.

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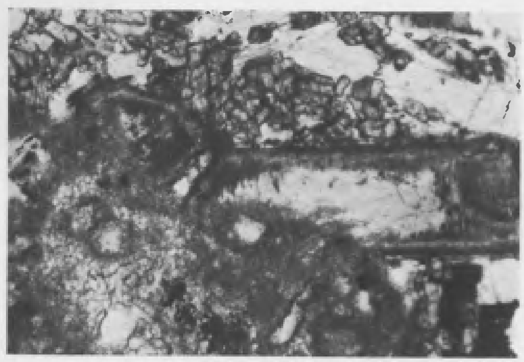

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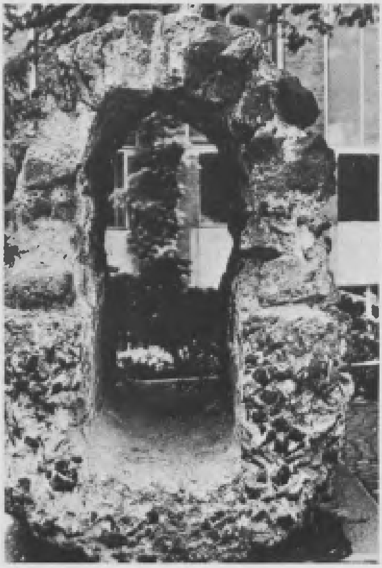
Figure 2. Lime-pozzolana concrete

A. Concrete walls of the cistern of Kameiros, Rhodos, ca 500 B.C.
B. Section of concrete in the cistern of Kameiros
C. The town wall of Ampurias, Spain, 50-0 A.C.
D. Example of lime-silica reaction in ancient concrete (harbour of Caesarea), (Malinowski et. al., 1961)



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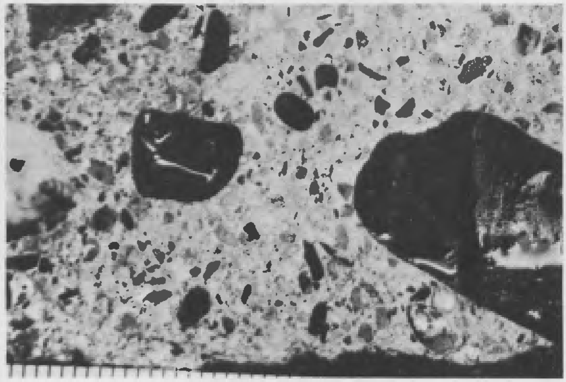

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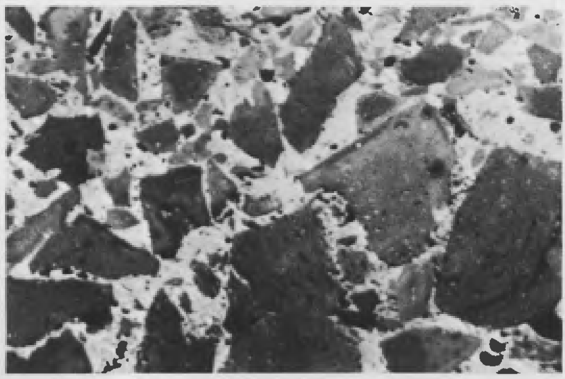

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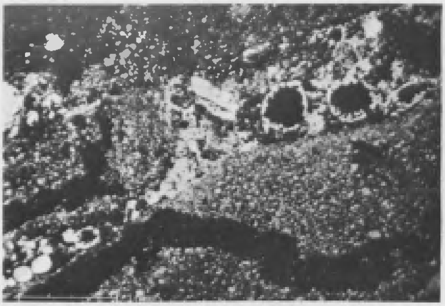
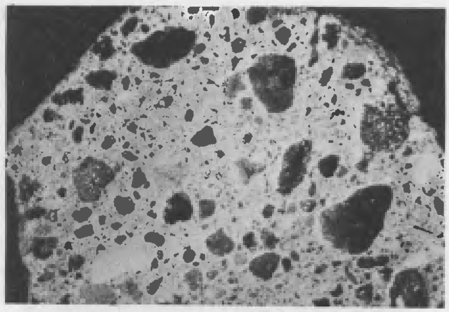
Figure 3. Different types of aggregates in Roman lime concrete.

A. Basaltic aggregate in the concrete of the Eifel aqueduct, Cologne, West Germany (100 A.C.)
B. Gravel concrete of the pavement substrate, Italica, Spain (150 A.C.)
C. Crushed brick - gap grade, Trier, West Germany (200 A.C.)





A

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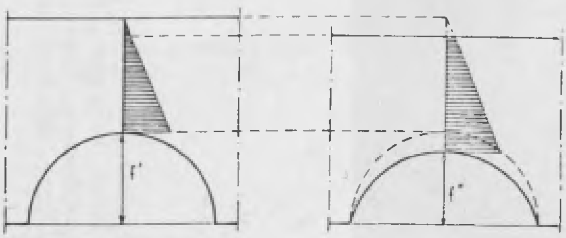
Figure 4. Ancient air entrained and lightweight aggregate concrete.

A. Air entrained concrete, Barbegal aqueduct - Alps, 0-100 A.C. (Idorn, 1959)
B. Lightweight lava aggregate concrete, Rome.

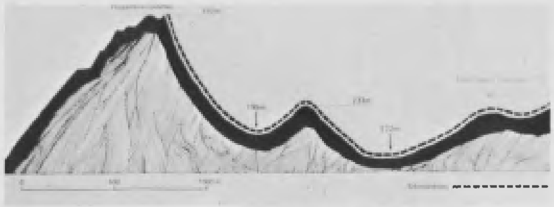


A

B

Figure 5. Scheme of pressure and deformations in the arch a) at early age and b) after long time loading (Malinowski, 1979[1])

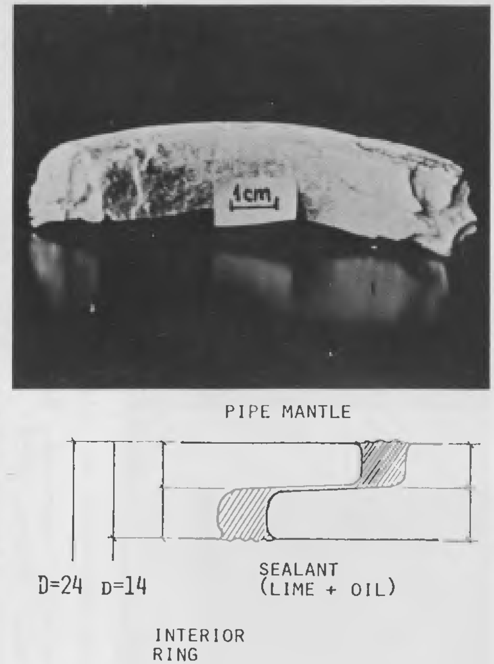
Figure 6. Opus insertum of gypsum concrete in the harbour walls of Kition, 600 B.C. (?)



A

B

C



D

Figure 7. Sealing of pressure aqueducts.

A. The lead aqueduct of the citadel of Pergamon, 200 B.C.
B. The lead pipe sealed in a stone block head, Pergamon, 200 B.C. (Fahlbusch - Malinowski, 1981)
C. Pressure conduit embedded in concrete, Alhambra, Spain, 0-50 A.C.
D. The sealing paste from a clay pressure duct, Rhodos, 500 B.C.

ANALYSE ET CARACTERISATION DE QUELQUE TYPE D'ANCIENS MORTIERS ORIENTALS

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SUMMARY

The aim of this experimental work is to find a correlation between the porous structure and the chemical-mineralogical composition of some ancient mortar.

The measurements habe been carried out on mortar from the minaret of Bahram Shah (Ghazni, Afghanistan).

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INTRODUCTION

Ce travail a le but d'étudier à fin de conservation les matériaux constitutifs du Minaret de Bahram Shah (1115 - 1149), situé dans une aire archéologique près de la ville de Ghazni, Afghanistan. Les investigations sur les matériaux (qui ont été executées pour le compte de l'ISMEO) étaient justifiées par le fait que les différents éléments du monument présentaient des conditions de perte de cohésion très variées, dues, peut-être, à leur différente composition et aux conditions extérieures (Galdieri, 1979). Le minaret a une hauteur de 40 mètres environ, il présente un affût à section stellaire à huit pointes qui s'appuye sur une base cylindrique, haute 2 mètres environ, érodée en plusieurs points par les agent atmosphériques. La structure portante est constituée de poutres en bois croisées en sorte qu'elles forment une structure stellaire et revêtues de mortier. Les parois estérieures sont décorées de stucs et de terres cuite en relief fixés à leur place par du mortier au gypse. Le monument ne présente pas des problèmes particuliers du point de vue staticité, mais le mortiers utilisés pour le fixage des claveaux de la structure portante et pour l'adhésion des panneaux décoratifs montrent un tel degré de perte de cohésion que l'adhésion même des éléments interessés n'est plus garantie. La caractérisation chimico-minéralogique des différents mortiers échantillonés, qui était fondamentale pour la définition d'une intervention conservative, s'est révélée utile afin de comprendre les causes de la baisse cohésion. Ce travail a le but de chercher une corrélation entre la composition chimico-minéralogique des mortier et leur structure poreuse, afin d'essayer d'expliquer leur différents états de cohésion.

ESSAYS

On a testé plusieurs échantillons de mortier extraits de différents parties du monument. En particulier:

Echantillon n°. 1 - base: mortier du revêtement des poutres;

" " 2 - première section, au-dessus du bandeau décoratif: mortier du bandeau décoratif;

" " 3 - deuxième section, deuxième page: mortier extérieur entre les briques;

" " 5 - deuxième section, façade Nord Est: mortier entre les briques;

" " 6 - base: mortier de restauration appliqué entre 1910 et 1920;

" " 8 - premier bandeau décoratif, troisième page Sud; mortier de la décoration;

" " 9 - premier registre: mortier entre la décoration et les briques.

Sur tous les échantillons on a executé les essais suivants:

- analyses de diffraction au rayons X pour déterminer la composition minéralogique;
- analyses chimiques quantitatives des ions sulfate et carbonate pour déterminer le rapport entre gypse et calcite;
- observations au microscope minéralogique pour la présence de grains calcaires;
- détermination de la porosité totale et de la distribution des pores pour évaluer la structure intérieure des matériaux.

RESULTATS

Analyses de diffraction aux rayons X

Les analyses ont été effectuées avec un diffractomètre Siemens Crystalloflex IV, radiation CuK_{α}. Les résultats des analyses, effectuées sur les échantillons finement moulus dans un mortier en agate et puis écartelés, sont reportés sur le Tableau I.

TABLEAU I

Analyses de diffraction aux rayons X

Echan.	Gypse	Calcite	Quartz	Feldspath	Muscovite	Chlorite	Aragonite
1	100		6				
2	100		11	9			
3	90	100	252	80	41	41	
5	100	82	161	69	35	27	
6	100		4	5			
8	15	100	25	19			20
9	100		10				

Les quantités de chaque composé ont été calculées par rapport à l'intensité du composé plus abondant (calcite ou gypse) avec fonction de liant.

Les analyses montrent une analogie de composition entre les échantillons nn. 1, 2, 9, qui se révèlent des mortiers constitués surtout de gypse, avec quantité variables de matériel inerte de nature terreuse. D'une manière analogue les échantillons nn. 3, 5 se révèlent de mortiers à liant mixte (gypse et chaux) avec de quantités variable de matériel inerte, dans ce cas un matériel argilleux ou, du moins, de la terre contenente des materiels argilleux. Ils s'écartent, parmi les mortier plus anciens, l'échantillon n. 8, qui résult composé de calcite et un peu de gypse avec matériel inerte de nature terreuse, et l'échantillon n. 6 qui, tout en ayant une composition analogue à celle des échantillon nn. 1, 2, 9, est un mortier de restauration posé dans les prémières années de 1900.

On peut voir que dans tous les mortiers testés les matériels inertes sont des composés de nature terreuse et, comme il résulte par l'observation microscopique, ils contiennent des quantités variables de grains calcaires à différents degrés de cristallinité, outre que des quantités variables de chaux carbonatée (échantillons nn. 3, 5, 6).

Analyses chimiques quantitatives

On a dosé quantitativement sur les mêmes échantillons de mortier soit le ion carbonate, soit le ion sulfate. On est parvenus à la détermination du ion carbonate suivant une methode volumétrique classique (Dimos, 1978), tandis que le ion sulfate a été dosé, en utilisant un Autoanalyzer Technicon II selon une méthode turbidimétrique (Tech. Autoan.), dans les solutions acqueuses résultantes de l'extraction des sels solubles avec de l'eau distillée. Les résultats obtenus sont reportés sur le tableau II, et sont enoncés comme % en poids du ion et comme % en poids de l'espèce chimique de laquelle le ion est répresentatif. Toutes les valeurs se réfèrent au poids humide.

TABLEAU II

Dosage quantitative des ions carbonate et sulfate

Echantillon	% $CO_3^{=}$	% $SO_4^{=}$	% $CaCO_3$	% $CaSO_4 \cdot 2H_2O$
1		54,16		97,08
2		54,16		97,08
3	12,90	7,63	21,50	13,67
5	11,40	12,32	19,00	22,09
6		30,99		55,54
8	53,10	2,98	88,50	5,34
9		37,78		67,72

Distribution du volume des pores

La mesure de la distribution du volume des pores a été effectuée avec un porosimètre à mercure CARLO ERBA Series 1.500, avec lequel on peut mesurer le volume des pores ayant un diamètre compris à peu près entre 0,01 microns et 100 microns.

Les mesures ont été effectuées suivant la methode récommandée par le Comité Italien NORMAL (Normal, 1980). Elles ont été effectuées sur de petits fragments, obtenus des échantillons originaux extraits du monument, de forme et dimensions pareilles (à peu près des cubes compris entre les tamis DIN. n° 3 et n° 5).

Chaque mesure a été effectuéee sur un seul fragment.

Les résultats des analyses porosimètriques, reportés sur le tableau III, sont les moyennes relatives à trois mesures. Ces résultats sont exprimés comme:

$$P\% = \text{porosité ouverte cumulée} = V_c^{max} \cdot \rho_a \cdot 100 \text{ où}$$

ρ_a = masse volumique apparente de l'éprouvette (g/cm^3)

V_c^{max} = volume maximal de mercure pénétré, par unité de masse, dans l'éprouvette (cm^3/g).

La distribution du volume des pores est exprimée en V% correspondant à pores avec diamètre compris entre intervals donnée.

CONCLUSIONS

On peut effectuer l'analyse comparative des résultats expérimentaux obtenus, sur la base du Tableau IV, dans lequel on reporte les valeurs comme pourcentage en poids de la calcite et du gypse contenus dans les échantillons, la porosité ouverte cumulée P% et les pourcentages des pores ayant un diamètre compris entre trois intervaux significatifs. Sur le tableau IV les échantillons sont cataloguée en ordre décroissant de concentration de gypse. L'échantillon n° 6, qui est un mortier de restauration recémment appliqué, est consideré comme le point de repère, soit quant à la technique d'execution, soit pour évaluer les modifications dues au vieillissement naturel des échantillons anciens.

Il est intéressant à remarquer que les échantillons nn° 1, 2, 3 et 5 révèlent une analogie de composition chimique outre que minéralogique. En plus, l'analogie de composition, qui peut être mise en rapport avec une analogie entre les pâtes ou les technologies employées, semble coincider avec le différent emploi des mortiers: les mortiers usés pour le fixage des décorations sculptées sont des mortiers au gypse (échantillons nn° 1 et 2), ayant un temps de prise très rapide; pour les mortiers employés pour le fixage des claveaux de la structure portante on a utilisé, au contraire, des pâtes avec un liant mixte de

TABLEAU III

Distribution du volume des pores

Echantillon	1	2	9	5	3	8	6
ρ_a (g/cm^3) ± 0,02	1,15	1,14	1,32	1,54	1,63	0,92	1,64
P% ± 1	58	54	49	45	43	71	36
V% avec d < 0,05 µm	10	7	11	12	16	12	21
V% avec 0,05<d<0,1 µm	2	2	1	3	6	8	7
V% avec 0,1<d<0,2 µm	1	1	3	7	3	10	7
V% avec 0,2<d<0,4 µm	3	1	1	4	5	10	7
V% avec 0,4<d<0,6 µm	2	0	2	4	5	7	2
V% avec 0,6<d<0,8 µm	2	1	1	5	4	8	2
V% avec 0,8<d<1 µm	3	1	1	5	4	9	2
V% avec 1 < d < 2 µm	18	12	15	38	24	36^{++}	14
V% avec 2< d < 4 µm	29	24	28	15	20		14
V% avec 4<d<10 µm	14	51^{+}	21	7	13^{+}		11
V% avec d > 10 µm	16		16				13

\+ V% de pores avec d > 4 µm
++ V% de pore avec d > 1 µm.

TABLEAU IV

Echan.	$CaSO_4 \cdot 2H_2O$ (% en poids)	$CaCO_3$ (% en poids)	P%	V% avec d<0,05 µm	V% avec 0,05<d<1 µm	V% avec d>1 µm
1	97	-	58	10	13	77
2	97	-	54	7	6	87
9	68	-	49	11	9	80
5	22	19	45	12	28	60
3	14	21	43	16	27	57
8	5	88	71	12	52	36
6	55	-	36	21	27	52

gypse et chaux, avec une quantité plus relèvante de materiel inerte, pour augmenter, peut-être, le temps de prise afin d'obtenir un produit final plus compact et résistant (Mariani, 1976).

En observant le tableau IV, il est surtout à souligner que les couples d'échantillons nn° 1, 2 et 3, 5 montrent des distributions porosimètriques équivalentes, en conformité avec leurs compositions chimiques et minéralogiques pareilles.

Après l'analyse des résultats rélatifs aux échantillons nn° 1, 2, 9, 3, 5 il semble qu'on puisse affirmer qu'une augmentation du pourcentage de gypse corréspond à une augmentation de la porosité P% et du pourcentage de pores grands. En effet, une augmentation du pourcentage de gypse de 14 à 97% environ, corréspond à une rélative augmentation de la porosité ouverte cumulée P% de 43 à 56% environ et du pourcentage de pores grands de 57 à 82% environ.

La confrontation entre les résultats concernant le mortier de restauration (échantillon n° 6) et ceux qui concernent l'échantillon n° 9, de composition pareille, nous permit de formuler l'hypothèse que les mortiers originaux ont subi un procès de dégradation du aux facteurs météoclimatiques de la zone, tels que, surtout, les fortes amplitudes diurnes et saisonelles. En effet, lorsque le mortier de restauration n° 6 a une porosité cumulée de 36% et une pourcentage de pores grands de 52%, le mortier ancien n° 9 nous donne, respectivement, des valeurs de 49% et de 80% environ. La présence d'une certaine quantité de calcite (échantillons nn° 3, 5) semble modifier la distribution porosimètrique, puisque la première fait augmenter remarquablement le pourcentage de pores moyens (ceux qui ont le diamètre compris dans l'interval 0,05µm - 1 µm). Cela semble s'accorder avec la distribution de l'échantillon n° 8, constitué essentiellement de calcite. En effet, dans ce cas la distribution porosimètrique est remarquablement plus plate avec un pourcentage plus élevé de pores moyens(52% environ). Il est à souligner, à ces propos, la présence dans lèchantillon n° 8, de petites quantité d'aragonite (une phase polymorphe de la calcite) la formation de laquel le pourrait être attribuée à cycles de solubilisation et récristallisation de la calcite dans les particulières conditions ambience où le monument est situé (Schippa, 1971).

Si on accepte cette hypothèse, on peut attribuer la valeur élevée de la porosité ouverte cumulée P% (71% environ) à des phénomènes de solubilisation interne du carbonate de calcium qui est le liant du mortier.

On peut enfin dire que la distribution porosimètrique des mortiers analysés varie en fonction de la composition chimico-minéralogique des mortiers mêmes. Il faut toutefois déclarer que les résultats obtenus devraient être considerés comme indicatifs parce que le nombre d'echantillons analysés est bas. On pourrait en effet obtenir des corrélations plus dirèctes entre la composition chimico-minéralogique et la distribution porosimètrique sur une base rigoreusement statistique, si on augmentait le nombre des échantillons analysés.

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RESEARCH ON LIMES AND INTONACOES OF THE HISTORICAL VENETIAN ARCHITECTURE.

Characterization of some "Marmorino" intonacoes from the 16th to the 17th century

Guido BISCONTIN* – Mario PIANA – Gianna RIVA*****

SUMMARY

This study shows the first results of an experimental analysis undertaken on "Marmorino" specimens extracted from buildings of the 16th and 17th centuries in the historical centre of Venice.

The study, which will be subsequently extended to a larger number of samples, has been carried out in an interdisciplinary way on the basis of historical researches and documents of the State Archives and is neant to suggest a suitable method for a physical, chemical and mechanical characterization of intonacoes from various historical periods.

The results obtained have shown how the intonacoes were made with a specific technological knowledge of the nature of the materials used.

The composition of the layers and their succession (for double-layer intonacoes), show how they have been particularly studied to create conditions of compatibility and continuity on the intonaco-masonry system.

Further tests will be made on the brick-intonaco system for a better understanding of the behaviour of both brick- and intonaco and their degree of compatibility.

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INTRODUCTION

This work constitutes a first contribution to the study of Venetian intonacoes of different historical periods, based on a scientific method that is intended to be applied later on, on a larger scale of wall facings. Although this first research represents a limited contribution, it is however thought that these findings are of some interest and therefore their publication may be useful.

The progressive and in some cases complete disappearance of the materials used in the Venetian masonry, together with the progressive loss of ability to work according to traditional technical methods on the part of the skilled workers, can cause deep and unacceptable changes of the town's image in the actual urban appearance as has already happened, for instance, to Burano. The renouncing of the materials and traditional working techniques in reconstructing external masonry facings and their replacement with materials and standardized techniques has produced other disadvantages besides the aesthetical ones.

The use of intonacoes provided with poor compatibility with the masonry has, in fact, caused a series of conditions of generalized decay with the consequent reduction of the endurance of the recently made intonacoes (lasting an average of 10 to 15 years), the increment of total costs of maintenance as well as acceleration of the decaying process of the masonry. In addition to this, there are the serious actuality of the problem of external masonry facings in the Venetian lagoon and the urgency to prepare new operative techniques and suitable material that can provide solutions in the lagoon. [1] [2].

It is thought that the undertaken study provides sufficient scientific elements for the characterization of the primary physical, chemical and mechanical properties of the historical intonacoes of determined historical periods consequently contributing to the knowledge of the historical evolution of the constructive techniques used in the Venetian historical centre [3].

HISTORICAL NOTES ON THE "MARMORINO" INTONACOES IN VENICE

The origin of the "Marmorino" intonaco, undefinable with accuracy (1), is placed

(1) The first known document in which a "Marmorino" work is possibly considered, is a factory contract for the Nuns of S. Chiara of Murano in the year 1473 (Murano Civic Museum - envelope 23 - transcribed and published by P. Paoletti in "L'architettura e la scultura del Rinascimento in Venezia", Venezia 1893) wherein refering to the masonry of the church to be built, it is pointed out that: ". . . ditj murj tutj belizar smaltar e terazar si dentro come de fuora . . .". The "Terazar" meaning the execution of a underlayer in ground brick, which is a typical characteristic of "Marmorino" and such a process is not present in any of the discoveries of Veneto-Byzantine or Gothic

around the middle of the 15th century, when, on the part of the Venetian skilled workers, begins the elaboration of a new type of external facing of the masonry in keeping with the technical and aesthetical requirements suggested by the classical ideal brought to the Venetian lagoons.

Around the middle of the 16th century, the "Marmorino" already shows its specific characteristics and qualities (2). These remain almost the same during the next four centuries making of it the only intonaco used in the town, especially in the 17th and 18th centuries.

Nevertheless, the aulic architecture of the Renaissance up to the 2nd, 3rd decade of the 16th century (3) and the minor architecture, for the whole century, are coated in the majority of cases with intonacoes that repeat the gothic decorative models (4).

The "Marmorino" is an intonaco made in a single-layer, with the exception of rare examples in more layers, composed of air-hardening-lime and fragments of stone materials, usually Istria stone. However, in some cases, the inert can be different. To the Istria powder other marbles such as "Saccaroide" (as in the analyzed specimen of the Vendramin Calergi Palace), or powders obtained by the grinding of glass (the so called "Granzolo") can be added.

The "Marmorino" was made by applying the lime directly to the masonry or, more

intonacoes.

(2) Alvise Cornaro, in one of his writings of about the year 1560, suggests that in the town of Venice, between the "Dogana" (Custom's House) and the Giudecca island, should be built a "Theatre in big stone, not of chisel stone, but of "Cotta", that will cost less than half the price and will be as durable as chisel stone, because the "Cotta", now that stucco has been discovered, it will be stucco coated and as can be seen, such stucco becomes stone as it is made of stone . . ." (". . . theatro di pietra grande, ma non di pietra da scarpello ma di cotta, che non costerà la metà e sarà opera durabile come di pietra da scarpello, perché la cotta hora che si ha trovato il stucco se istuccherà e, come si vede, tal stucco si converte in sasso perché è fatto di sasso . . ." - Archivio di Stato di Venezia, Savi ed Esecutori alle Acque, b. 986, filza 4). As can be clearly understood, we are dealing with an intonaco (the Cornaro's "stucco" being in fact "Marmorino") of recent formation, but also sufficiently experimented if its application is with no doubt suggested on a public work of such significance.

(3) For example the intonaco of the second cloister, in the S. Giorgio Maggiore convent, of which during recent restoration work, some traces have been found. This intonaco of the late 15th century period, of a single-layer of lime and marble-powder, is a typical intonaco of transition, in fact, the white "Campitura" in renaissance style, joins a strip of under-eaves, fresco-painted with floreal motives of formal gothic tradition.

(4) Two exemplar cases are mentioned: the first, in the S. Marco quarter, at S. Luca; it is an intonaco conserved nearly intact on a building of the 16th century, white "Campitura" and fresco painted with "Marcapiano" strips contouring the windows, protected by several layers of an oily substance (probably linen oil). The second case is an intonaco, fairly well conserved, present on the façade of a habitative unit belonging to a block of buildings of the 16th century in Corte Nova at Castello, with a single-layer intonaco, fresco painted on the whole surface with a decorative motive consisting of a two coloured brick-work displayed in diamond shapes, that recalls the formal example of the stone facing of the Ducal Palace.

often, on a layer of ground "Cotto", bounded with air-hardening-lime ("Cuogolo" lime) of semi-hydraulic type ("Negra" or "Brovada" lime) (5).

The extreme compactness and durability that characterize the "Marmorino" intonaco and distinguish it from other intonacoes of the same composition, are due both to the specific attention in the choice of the binding and to the extreme care in the execution and final protective treatments.

In all contracts between the 16th and the 18th century pertaining to works in "Marmorino" the repeated washing-down of the masonry and the perfect workmanship were considered as essential, in fact these are the basic conditions for a good result. The repeated washing-down ("Bagnatura") of the masonry before, during and after the formation of the intonaco, creates the optimal conditions for a slow drying hold of the binding necessary to the arduous and lenghty metal spatula work, protracted till the drying up of the mortar. The continuous going over the surfaces with steel-wool determines a progressive compacting of the intonaco, by adjustement of the individual grains of the inert and by extraction of the water present in the mortar which is by this process brought to the surface. (The word "pacciarina", which means the water extracted, is still remembered today).

Once the intonaco has dried-up, it is treated with "Saponata", to which a further wax treatment is often added (6), or by applying one or two coats of linen oil (7), this

(5) In Venice, the use of this particular kind of lime starts in the first decades of the 15th century, with the conquest of the Paduan territories. The "Serenissima" since then has had access to the Colli Euganei Caves, the zone of the marl stone which is extracted for the production of this lime and has hydraulic characteristics.

(6) Hereunder is quoted a passage of a treatise of the 17th century on the executive technique of the "Marmorino": ". . . e detta smaltatura deve essere benissimo lisciata con la cazzuola fino a tanto che s'indurisca, poi si piglia sapon damaschino e distemperassi nell'acqua a modo di liquido bianco per imbiancare i muri, e con quello si vadi con il pennello spianzando la smaltatura un poco per volta, e cosí spianzata di fresco si vadi con la cazzola lisciando con diligentia. E quando tutta sarà lisciata, si lasci impassire, o poi piglissi un panno di lino e freghissi benissimo, poi piglissi cera in formelle e con quella in cambio di cazzola si vadi benissimo con diligentia per tutto lisciando, e poi freghissi un'altra volta con panno di lino". (G. Viola Zanini, "Della architettura...", libri due, Cadorino, Padova I, 1629). ("And the said covering must be smoothed down very well with the trowel until it becomes hard. Damascus soap is then taken and distempered in water until it becomes a white liquid in order to whiten the walls. The surface must be splashed a little at a time with a brush soaked in this liquid. While still freshly damp it must be smoothed down with the trowel diligently. When it is all smooth, it must be left to dry, and then rubbed down with a linen cloth and later rubbed down with solid wax and a linen cloth").

(7) Also see from an example of the 17th century; in a contract which reads: ". . . Far tutte le terazadure di foravia di detta fabrica di due mano di terazo biancho, benissimo slisutta, bagnando benissimo le muraglia e dandoli poi il suo oglio di lino . . . Far li camini dalli copi in suso tondi, . . . et questi smaltati, cioè interazzatti di terazo rosso e poi slissati di terazo biancho . . . , dandoli il suo oglio de lino stabelliti di tutto ponto" (Archivio della Confraternita di S. Giorgio dei Greci, Armadio B, colto IV, n. 55, 1658). (". . . Make all the 'terazzare' of the outside of the

being a very ancient practice in Venetian Masonry (8). These applications are made for aesthetical reasons, trying to imitate as much as possible a smooth shiny stone, and for protective reasons to produce a consumable surface.

Particular care was taken in making the intonaco on the walls more exposed to the inclement weather by using, at times, additives that increase the permeability of the binding, such as metal grit, the so called "Marogna" with a high content of silicon and magnesium.

In some instances the "Marmorino" was beaten with a particular pointed instrument, in order to reproduce the "Martellina" effect of the Istria stone (9) or, to the mixture were added coloured loams (clays); sometimes the ground "Cotto" was used as inert when making strips, usually in relief, for framing the white inner panels ("Campiture") (10).

building in two layers of white 'terazo', beautifully smooth, wetting the brickwork very well and passing over it afterwards with linen oil . . . Make the chimneys round from the roof and varnished, that is, made of red 'terazo' . . . and then smoothed down with white 'terazo' giving them linen oil to make them beautiful to perfection").

(8) In the Gothic period, the practice of applying layers of dehydrating oils to which were added cementing pigments not only on the limewashes or directly on the masonry (see for example the treatment of the external façades of the Frari Church at S. Polo) but also to the external stones of the buildings. Enlightening this example is the contract dated 1431 regarding the decoration of the façade of the "Ca' d'Oro" on the Grand Canal; transcribed and published by B. Cecchetti, "La facciata dalla Ca' d'Oro", in Archivio Veneto, XXXI, pagg. 203-204, 1886: "Apresso vuol che tuta la corona con i archeti e con le chuete che sera datorno i diti archeti e la chornisse che possa i diti archeti suso sia tuto dado de biacha a oio e che tuti i merli sia ombrizadi a muodo de marmoro e cum qualche segno negro atorno i ori de i diti merli se lo i parera. E per lo simel vuol esser tochado de negro el campo de le chuete va entro i archeti. Apresso vuol che tute le piere rosse che se in la dita fazada e tute le dentade rosse sia onte de oio e de vernixe con color che le para rosse. Apresso vuol che tute le ruosse e vide che se entro la dita fazada sia tute dade de biacha a oio e penzer i campi de negro a oio per muodo chel stia ben. E per lo simel vuol esser impechado i campi de tute le foie de la cornisse del primo soler tute con el voltar de i cantoni e con quatro piane che se in la calle de negro a oio."

(9) The examples of the limework of the Gesuiti Church in Cannaregio are quoted, and particularly the specimen conserved from the Gesuati Church on the Zattere.

(10) This is the case of the external intonaco of the "Scuola Grande di S. Giovanni Evangelista" at S. Polo with intonaco of white "Specchiature" contoured by grey stripes in relief, probably made in the year 1731. Examples of the use of strips with ground "coccio" used as inert are the analized specimen of the 16th century from Ognissanti Church of Pellestrina and the 18th century intonaco from the bell-tower of S. Zan Degolà in S. Croce.

ORIGIN AND DESCRIPTION OF THE INTONACO SPECIMENS SUBMITTED FOR EXPERIMENTAL INVESTIGATION

The intonaco specimens studied and submitted for physical and chemical investigation come from the "Marmorino" group of intonacoes belonging to hand-made buildings dating from the 16th century to the 18th century.

Ca' Vendramin Calergi (XVI century) – started in the year 1482 by Mauro Codussi

Three specimens of "Marmorino" intonaco have been extracted from different heights of the wall facing north-west.

Specimen N. 1: Height of extraction 7.5 meters above medium sea level. [Fig. 1 - 2]

The inner layer, in conctact with the brick, shows a fairly regular thickness of 3.5 mm of a pink colour, due to the obvious presence of powder and traces of ground brick.

To this first layer adhers a second one of the same thickness, white in colour and composed of fragments of Istria stone and granules of microcrystalline marble (0.5 ÷ 1 cm) bound together by lime.

The outer surface presents itself with a differential alteration caused by the heterogeneity of the materials employed (marble and binding). The decay is apparently of a traditional type (11) and is limited to the outer layer.

Specimen N. 2: Height of extraction approximately 14.0 m above medium sea level.

The intonaco presents itself with characteristics similar to those described for specimen N. 1. The thickness of the inner layer is approximately 4.5 mm, while the outer layer is approximately 2.5 mm. The latter is composed of fragments of Istria stone and marble, measuring an average of between 0.5 and 1 cm.

The surface of wear is in some places still well conserved.

Specimen N. 3: Height of extraction 9.00 m above medium sea level.

Characteristics similar to those of specimen N. 2, thickness of inner layer 5.0 mm; outer layer 3.5 mm.

The multiple system in two layers of the Vendramin Calergi Palace (specimens N. 1, 2 & 3) has probably been made to assure the continuity of the system in respect to the physical and mechanical properties of the masonry, thereby achieving a compatibility among the various layers as to the principal cyclic phenomena to which the masonry is exposed, i.e. thermical expansion vapour permeability, gases etc.etc. The inner layer in particular, acts as a cushion between the outer layer

(11) The main factors of decay are: mechanical wear of wind and dusts, washing away and also the chemical aggressivity of the air.

and the masonry, preventing the formation of surfaces of discontinuity on which, as is known, local mechanical and later physical phenomena of decay could take place.

The specimens, although the composition of the layer is evident, have a good adherence between themselves and the masonry (12).

Ognissanti Church, Pellestrina (ca. 1535)

The church, of an old foundation, was enlarged in the year 1535 and consecrated in the year 1536, and in all probability the reconstruction of the bell-tower and the limework date back to that period.

At the beginning of the year 1600 the church was enlarged and elevated, incorporating into the roof of the church part of the brick work of the bell-tower. The specimen extracted from this zone is in good condition of conservation.

Specimen N. 4: Height of extraction approximately 20.0 m above the medium sea level, facing north-west. [Fig. 3]

The single layer intonaco mixture of a pink colour, extracted from one of the contour lines of the white panellings, is composed of brick powder, coarse at times (with a thickness up to 1 mm) with the presence of several white nodules of lime, mainly combined together. The outer surface is accurately smooth and still well conserved, thanks to the work done by spatula which has conferred to it a notable resistance to abrasion.

To a naked eye inspection the surface does not seem to suffer from visible phenomena of decay.

Carminati Palace (XVII century)

Architecture of the middle of the 17th century.

Specimen N. 5: Height of extraction approximately 5.50 m above medium sea level. Facing: north-west. [Fig. 4]

Presents itself with a single layer, white coloured and a thickness varying from 6 to 8 mm; composed of a mixture presumably of lime and notable quantities of fragments of Istria stone in splinters of dimensions up to 5 ÷ 6 mm.

The outer surface has degraded and it has lost its original smoothness (13).

Spanish Synagogue (XVII century)

The building was realized by Baldassare Longhena in the year 1635 and was the readaptation of a previous construction dating back to the year 1584.

(12) The extraction of these specimens was undertaken manually, applying a certain amount of physical force in removing the intonaco crust.

(13) The analyzed specimen has been extracted from a particularly degraded zone, while in other zones the intonaco has conserved its original smoothness.

Specimen N. 6: Height of extraction 6.00 m above medium sea level.

Facing: north [Fig. 4]

The intonaco presents two layers with the same characteristics as specimens N. 1, 2 & 3.

The inner layer, of a pink colour, has a thickness varying from 5 to 7 mm. The outer layer, coloured white, has a thickness of 1.5 to 2 cm and is composed of a mixture of chips of Istria stone with a granulometry of 0.3 to 0.4 cm. The surface is well conserved, but presents some degraded parts, as specimens N. 1, 2, 3 & 5, while in other parts it retains the original surface smoothness.

EXPERIMENTAL PART

The tests made on the series of intonacoes previously described in a macroscopic way have been of a chemical and physical nature. Testing such as X fluorescence, chemical analysis, determination of vapour permeability, S.E.M. observation and H_2O absorption have been made on the specimen.

Other testings, as the determination of soluble salts and their quantitative analyses, the porousity, presence of organic substances such as oils, the granulometry and the vaporization speed, are part of the general research and are in the execution stage.

X Fluorescence

The analysis made with an ORTEC TEFA 6111 instrument, is of the qualitative nature and has the object of defining the field of quantitative analyses. Following this it is planned to undertaken quantitative X analyses by using a series of standards of intentionally studied compositions [4]. The results obtained by the analyses are listed in Table N. 1.

From the X fluorescence analysis it can be noted that the elements that are present in a greater quantity reflect compositions of recurring use in intonacoes. There are however significant indications:

a) as regards the "Marmorino" intonacoes in two layers the constant presence of iron in a good quantity on the first layer can be noted, due to the presence of brick being grinded and added. The external parts are, on the other hand, almost entirely composed of mortar of lime aggregated mainly of $CaCO_3$ bases.

b) There are obviously many elements present in smaller quantities. This is due to materials employed and to the environmental effects. It is possible that metals such as Ti., Cr., and Mn. belong to the mixtures of the brick, while Cu. and Zn. may be due to external factors.

Table N. 1

Sample	Prevailing elements*	Secondary elements*
1 (int.)	Na., Mg., Al., Si., Ca., Fe.	S., Cl., K., Ti., Cr., Mn., Cu., Zn., Rb., Sr., Pb.
1 (ext.)	Mg., Al., Si., Ca.	Na., S., Cl., Fe., Sr.
4	Na., Mg., Al., Si., Ca., Fe.	Cl., Ti., Mn., Cu., Zn., Br., Rb., Sr., Pb.
5	Na., Mg., Al., Si., Ca.	S., Cl., Mn., Fe., Cu., Zn., Sr., Pb.
6 (int.-ext.)	Na., Mg., Al., Si., Ca., Fe	S., Cl., Ti., Mn., Cu., Zn., Rb., Sr., Pb.

* As is known, the X fluorescence analyses are qualitative, however they permit a certain differentiation of the elements when the quantities are clearly different.

c) The presence of Rb. and Sr. and in some cases of Sr. alone, is probably due to impurities present in the limes and therefore it is a quantitative analysis of these elements that will eventually supply useful indications to the origin of the limes used. The presence of Br. found in the specimen N. 4 taken from the bell-tower of Pellestrina, must be related to the nearness of the open sea.

d) Furthermore, it has been noted the diffused presence in almost all the specimens of small quantities of lead, could be related to the use of pigments of lead-based mixtures.

A deeper study of the problem is deemed useful as it has presented itself with more evidence in the analysis of intonacoes of lesser thickness (14).

Chemical analyses

The analyses have been carried out according to traditional methods: the Ca., Mg., and Fe. determinations have been made by means of atomic spectrophotometry of ab-

(14) On two presumed painted specimens from the church and the convent of the Frari (XIV century) X fluorescence analyses have been made: in both cases a rather consistent presence of Fe. and Pb. was shown. It can be deduced that these elements constituted the pigmental part of the crust. Determinations are being made to verify the possible presence of organic bindings of a greasy nature, such as oils, as suggested in historical archive records. This technique, employed on particular bindings which have this kind of coating, coloured with metallic oxides and of a thickness inferior to one millimeter, have resisted, even though in small areas up until the present day.

sorption.

On double layer specimens, analyses of each layer have been made after the mechanical division of the same. The results of these analyses are reported on Table N. 2.

Table N. 2

Sample	I.L. %	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	CO_2 %	CaO %	MgO %	Cl^-	$SO_4^=$
1 int.	25.7	29.2	3.6	2.6	21.8	21.1	11.3	+	+
1 ext.	42.8	7.7	0.6	0.4	42.2	42.9	6.8	+	+
2 int.	31.1	31.2	4.0	1.7	27.5	20.6	10.6	+	+
2 ext.	40.6	11.9	0.6	0.1	36.5	35.1	8.8	+	+
3 int.	28.4	28.9	9.1	1.6	24.7	19.7	9.5	+	+
3 ext.	40.8	5.0	6.9	0.2	40.5	35.4	11.4	+	+
4	30.8	25.9	4.9	2.6	23.9	24.4	10.5	+	—
5	42.3	1.6	1.4	0.1	39.5	48.5	3.2	+	+
6 int.	27.1	29.8	6.1	1.7	25.8	24.1	7.8	+	+
6 ext.	38.9	6.6	1.9	0.5	37.9	40.5	5.1	+	+

From these analyses, some indications emerge on the composition of marble-like intonacoes. The double layer marble-like intonacoes confirm what is macroscopically visible; i.e. the presence of the underlayer of brick particles of differentiated granulometry and lime with a consistent presence in binding in respect to present day compositions. In the outer layer only lime is present, always mixed with small pieces of marble or Istria stone. In all the specimens the presence of sulphate chloride has been noted.

– The one-layer "Marmorino" from Palazzo Carminati (specimen N. 5) is exclusively composed of lime mixed with small pieces of Istria stone.

– The single-layer intonaco from the Pellestrina bell-tower (specimen N. 4) has a composition similar to that of the double layer marble-like intonaco and is the only specimen in which sulphates are absent.

– Furthermore, it must be especially pointed out that the three specimens of marble-like intonaco of the Venetian Vendramin Calergi Palace (specimens N. 1, 2 & 3) have a high presence of Mg., existing also in part of the basic limes and probably due to the use of products of a certain dolomitic nature.

– The presence of brick-powder must be related to the resistance of the intonaco as it could have a hydraulic effect producing more resistant systems.

Vapour permeability

This test, of a physical kind, has been carried out on two specimens of marble-like intonaco, a single-layer specimen (N. 5) and a double-layer specimen (N. 6) using a method already employed for stone materials [5]. The study of vapour permeability is of particular importance as it must be related to the phenomenon of detachment of the intonaco from the substratum. The test, considering the insufficient disponibility of specimens, was carried out on very small surfaces (between 12 and 15 cm square) in comparison with the thickness of the specimens. The results, obtained at 20 °C are listed in Table N. 3.

Table N. 3

Sample		Thickness cm	Vapour permeability g/24 h m^2
5	1	0.6	306
6	2	0.6 ÷ 0.7 (int.) + 0.1 ÷ 0.15 (ext.)	378

As can be noted, the values of vapour permeability are sufficiently high and show a fair disponibility of the two intonacoes to be transversed by vapour.

It is interesting to note – although it must be confirmed by further tests – that vapour permeability is higher for the double-layer intonaco, even if this is thicker than single-layer intonaco. Naturally the values obtained must be put into relation with the permeability of the brick and the brick-work in order to supply possible indications as regards the adhesion to the brick-work of intonacoes made with less permeable materials.

Water absorption

The test has been carried out by immersing the specimen into distilled water for 48 hours and the results obtained from the three specimens are listed on Table N. 4.

Table N. 4

Sample	Water absorption after 48 h %
1 (2 layers)	17.5 ÷ 18.1
5 (1 layer)	14.9 ÷ 15.6
6 (2 layers)	16.9 ÷ 17.3

A greater value of absorption of H_2O for the double layer specimens (specimens N. 1 & 6) is noted in coherence with the results of the vapour permeability test.

A closer examination of this investigation could be represented in the future by a study of the curves of evaporation of the specimens and those of the brick-work.

Preliminary results of tests of evaporation on some specimens after the absorption of distilled water over a period of 48 hours have shown that the percentage of evaporated water in 4 hours is approximately 30% at a temperature of 20 °C. and the R.H. of between 60-70%. Corresponding tests carried out on brick-work specimens 1.0 cm thick under similar thermo-hygrometrical conditions have given inferior evaporation values inferior to between 15-20%.

CONCLUSIVE CONSIDERATIONS

The results of the tests, although still partial, show that the considered intonacoes were based on specific technical knowledge of the nature of the materials employed.

In fact it is verified that both the succession of the layers and their composition are particularly adapted to create compatibility and continuity conditions in the masonry-intonaco system.

The composition of the layers displays the presence of an intermediatory layer of a mixed nature between the outer surface and the brick-work. The probable function of this layer being that of a mediator between the properties of the connected materials.

As regards the single layer intonacoes it is noted that the composition of the mixture with the binding and the inert with a granulometry of the dimension of a few millimeters has most probably the function of making the system more transpirable.

The limited number of the specimens investigated and the limit of the tests have not permitted, as has previously been underlined, a complete interpretation and clarification of the problem. To this end physical tests are being conducted in order to create a better understanding of the phenomena of transpirability and adhesiveness (integral porosity and pore size distribution, granulometric analysis, velocity of absorption and the evaporation of water). Chemical tests will include the quantitative definitions of the components (impurities, soluble salts and a determination of eventual oils present, etc.). Also planned is a chemical and physical investigation of the system of brick-work-intonaco in order to establish the relationship between the two components and the grade of compatibility of the system.

From the total result of these investigations it is hoped to show a characterization on a scientific basis of the historical materials in relation to techniques and production technologies as well as the construction of various historical periods. It is also hoped to give some indications for the preparation of new intonaco for use on the masonry of the historical centre of Venice.

It must however be kept in mind, that the knowledge acquired cannot be readily trasferred to new standardisations, firstly because of the diverse change in actual atmospheric conditions in respect to the historical period for which they had been formulated and secondly because the historical masonry has suffered over the centuries undergoing many complex trasformations as compared to their original conditions.

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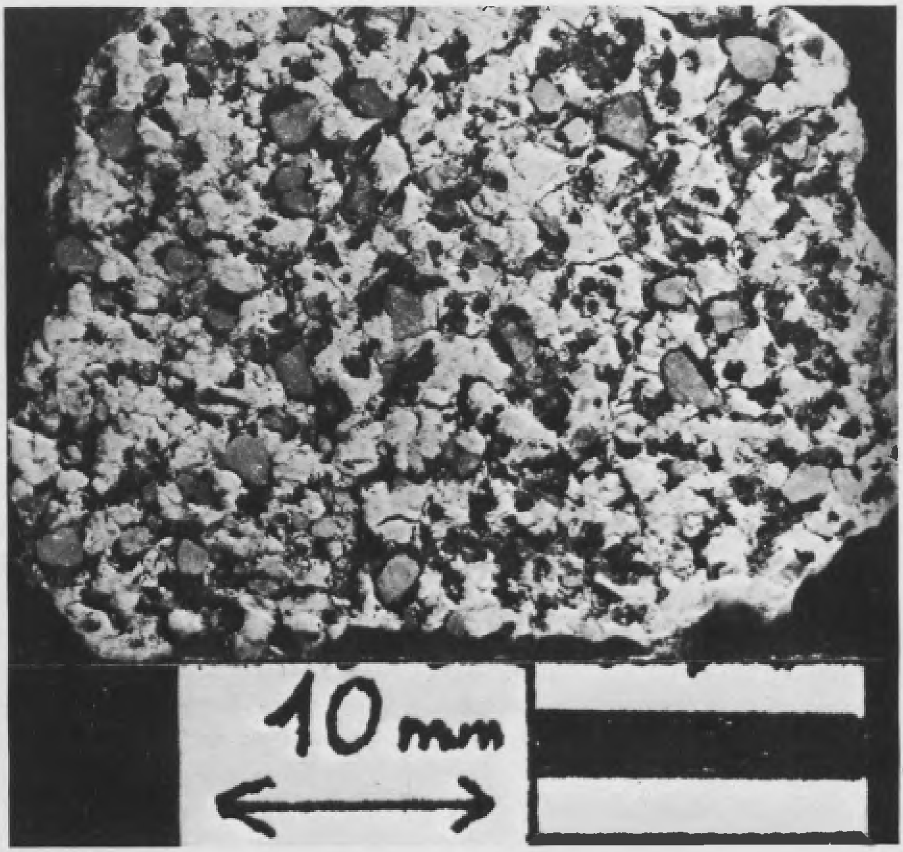
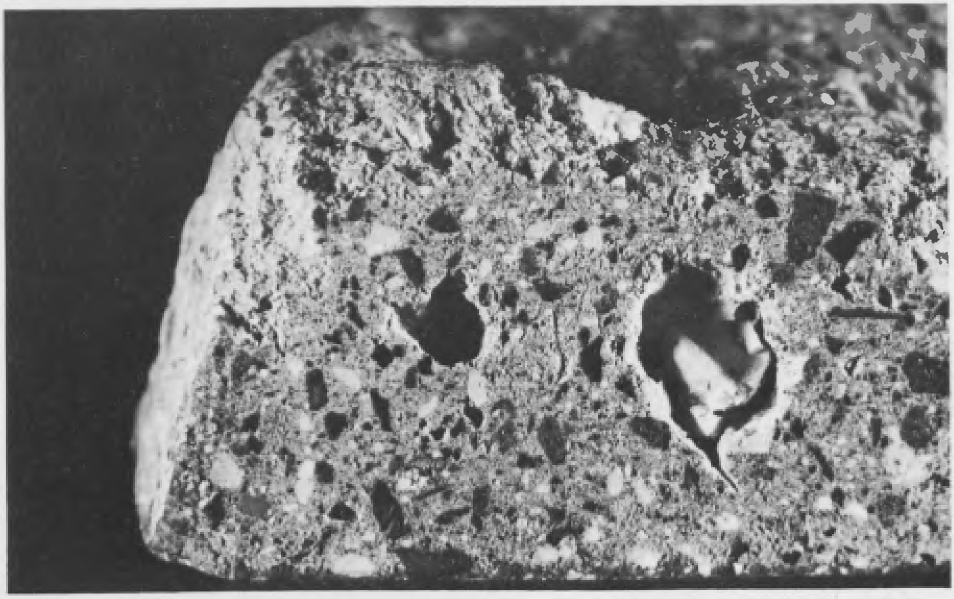


Fig. 1 and 2 -- Ca' Vendramin Calergi (XVI century). Section and view seen from above of the intonaco specimen N. 1.



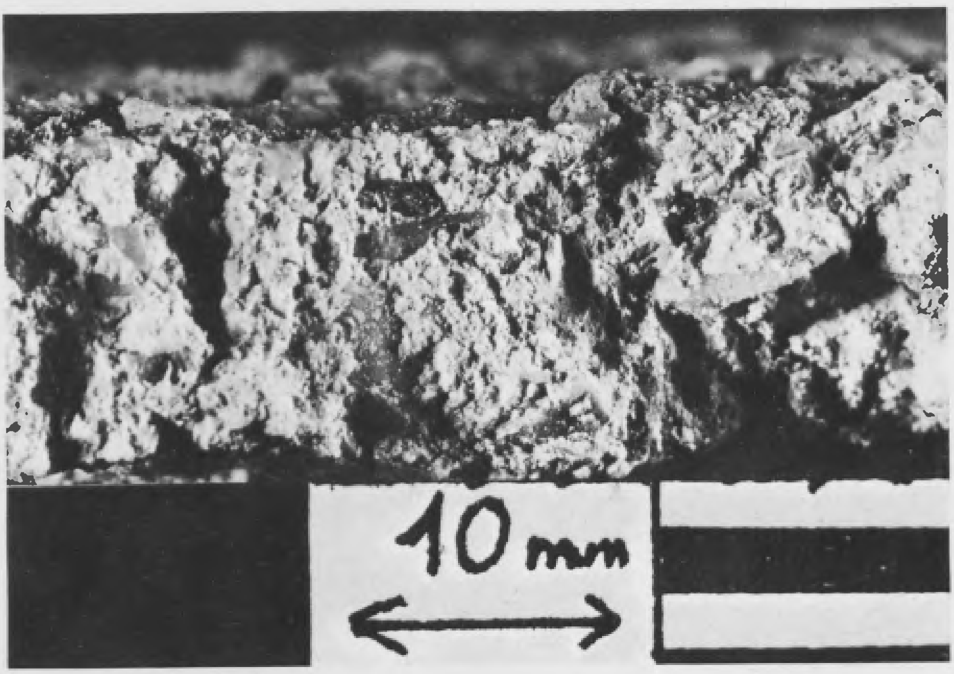


Fig. 3 — Ognissanti Church (ca. 1535). Section of the intonaco specimen N. 4.
Fig. 4 — Carminati Palace (XVII century). Section of the intonaco specimen N. 5.

Les matériaux des enduits traditionnels

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Summary

This text lists briefly costitutive materials and the composition of various mixtures used during the course of centuries for architectural plasterworks.
Cement and synthetic materials used recently for such works are excluded from this study.

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Les matériaux des enduits traditionnels

Introduction

Cette étude présente un exposé synthétique sur les matériaux constituants et sur la composition des différents mélanges employés depuis l'antiquité pour les enduits architecturaux.

La connaissance de l'utilisation de ces matériaux est le résultat d'une expérience acquise au cours d'années de travail sur les décorations murales externes et internes et qui est ici résumée dans ses lignes essentielles. Nous n'avons pas reporté les résultats des enquêtes morphologiques et des analyses chimiques et pétrographiques qui ont contribué à former cet ensemble de connaissances, mais les documents qui contiennent ces données peuvent être consultés à l'Istituto centrale del restauro à Rome.

Les principaux matériaux constitutifs des enduits destinés à recouvrir les structures architecturales ont été, au cours de l'histoire, l'argile, le gypse et la chaux avec ses différentes charges organiques et inorganiques. D'autres matériaux, dont nous ne parlerons pas ici, ont été également utilisés dans les enduits, plus encore comme additions que comme matériaux de revêtement proprement dits: sève de plantes, colles animales, sucre, excréments, etc.

Composition des mortiers

Le matériel de base pour la composition des enduits est le mortier. Il s'agit d'un mélange, à consistance plastique, de certains matériaux avec de l'eau, ayant la propriété de faire prise, c'est-à-dire de se solidifier et de durcir ensuite en adhérant aux matériaux de construction pour les lier entre eux et pour revêtir les surfaces.

Les matériaux agglomérants utilisés pour leur préparation, des origines jusqu'à nos jours, et dans toutes les régions du monde, ont toujours été les suivants: <u>terre argileuse, gypse et chaux</u>.

Ces liants peuvent être utilisés tout seuls, à condition que la couche de mortier soit assez mince; l'épaisseur de la couche doit diminuer au fur et à mesure que le coefficient de retrait augmente.

La chaux a un coefficient de retrait élevé, les terres argileuses un coefficient moyen et le gypse un coefficient égal à zéro.

Pour réduire le retrait des agglomérés à coefficient élevé et moyen, on y ajoutait des agrégats, c'est-à-dire des matériaux incohérents, organiques et inorganiques, avec des particules assez fines et constituant une armature interne et un squelette rigide pour le liant.

Les agrégats peuvent être organiques: paille, balle de riz et de blé, fibres de lin, soies animales, etc. mais aussi inorganiques: sable, poudre de marbre ou de pierre, tessons pilés et toute terre volcanique, comme pouzzolane, trass, terre de Santorin, etc.

Selon le liant utilisé on peut avoir des mélanges différents:
a) mélanges durcissant par perte d'eau: terres argileuses;
b) mélanges durcissant par absorption d'eau cristallisant avec le matériel: plâtre;
c) mélanges durcissant par réaction chimique et perte d'eau: chaux et ciments.

a) Mélanges durcissant par perte d'eau

Ce sont des mélanges formés par des terres argileuses qui constituent, des origines à nos jours, les revêtements les plus répandus. Les structures en cannes et herbes tressées, en bois et en terre étaient toujours couvertes d'enduits à base de terres argileuses, du moins dans une grande partie du monde: Europe, Asie, Afrique, Amérique du Nord et du Sud.

Dans ce genre de mélanges se trouvaient toujours des fibres organiques et inorganiques; les premières ayant la fonction d'armature interne pour en augmenter la compacité et l'adhésion à la structure, les deuxièmes (terres maigres et sables), comme nous l'avons déjà dit, pour en réduire le retrait pendant la phase de dessèchement.

Afin d'augmenter la cohésion de ces mélanges on y ajoutait (comme on le fait encore en Afrique et en Orient) des excréments animaux. Les enduits ainsi préparés recevaient un finissage superficiel qui pouvait être grossier, réalisé avec des brosses à mouvement circulaire, ou bien lisse et ensuite couvert d'une couche d'argile blanche (kaolin) ou de plâtre ou encore blanchi à la chaux.

b) Mélanges durcissant par absorption d'eau

Le plâtre et l'eau forment un mélange durcissant par cristallisation. Dans certaines formes hydratées ou anhydres, il constitue un mortier simple, pouvant être donc utilisé sans y ajouter des agrégats et présentant la caractéristique qu'il peut être façonné parce qu'il conserve un degré élevé de plasticité pendant un temps assez long, ce qui fait qu'il ne se déforme pas par son propre poids.

Ce mélange a été couramment employé sur des structures de terre argileuse ou pierre, de l'ancienne Egypte à la Mésopotamie jusqu'à la région sud-iranienne où il est toujours utilisé.

c) Mélanges durcissant par réaction chimique

Ces mélanges utilisent la chaux en tant que liant, qui est prédominante autour de la Méditerranée et que l'on obtient par calcination de pierres à chaux compactes, à structure microcristalline, extraites de rochers sédimentaires de carbonate de calcium et contenant des impuretés en quantités variables; leur pourcentage définit les différents types de chaux.

Si les impuretés - magnésium, silice, oxyde de fer,etc. - sont inférieures à 5% on obtient de la chaux grasse, tandis que, si les impuretés sont supérieures à ce pourcentage, on a de la chaux maigre et de la chaux hydraulique(1). En excluant la chaux maigre et hydraulique ainsi que les ciments, nous allons considérer uniquement les mélanges à base de chaux grasse.

La chaux normale grasse , obtenue par extinction de la chaux vive en mottes, a une consistance pâteuse et onctueuse au toucher, surtout si son degré de pureté est élevé (x). Elle peut encore être diluée avec de l'eau pour former du lait de chaux et de l'eau limpide de chaux. Avant l'utilisation, la chaux normale grasse doit mûrir pendant six mois au moins, même si les romains étaient obligés à l'employer seulement après trois ans. A présent, la chaux grasse se trouve également en poudre fine sous l'appellation de chaux hydratée. Par chaux hydratée on entend le produit obtenu à partir de l'oxyde de calcium ayant réagi, à l'aide d'un traitement particulier, avec la quantité stoechiométrique d'eau pour former de l'hydrate de calcium en poudre sèche.

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(1) Il ne faut pas confondre chaux hydraulique et chaux hydratée . Par chaux hydraulique on entend le produit obtenu par calcination, entre 1.000 et 1.300°C, de calcaires argileux et marnes avec 8 à 27% d'impuretés.
La chaux hydraulique était diffusée déjà au XVIème siècle, suivant les sources. ANDREA PALLADIO, I quattro libri dell'Architettura,Venise, Dominico de' Franceschi, 1570, livre I, chap.V, p.5: "Une fois cuite il faut la mouiller, mais sans verser toute l'eau ensemble, mais en plusieurs fois et continuellement pour qu'elle ne brûle pas et qu'elle soit bien détrempée. Après on la laisse dans un endroit humide et ombragé sans rien y ajouter , sauf du sable léger pour la couvrir.Plus elle sera macérée et plus elle sera tenace et meilleure (chaux commune), sauf celle faite à partir d'une pierre écailleuse, comme celle de

(x) En italien "grassello"

En ajoutant à la poudre la quantité juste d'eau on obtient la chaux grasse qui doit attendre au moins 24 heures avant de pouvoir être utilisée. Toutefois ce genre de chaux donne des mortiers de qualité inférieure. Les mortiers les plus résistants sont toujours formés à partir de la chaux grasse obtenue par extinction en fosse.

L'amélioration des propriétés de prise et de durcissement de la chaux grasse éteinte depuis longtemps, par rapport à celle éteinte récemment, est due, selon les dernières recherches, à une croissance progressive des cristaux d'hydrate de calcium.

Le processus de durcissement de la chaux est appelé carbonatation parce que pendant la prise l'hydrate de calcium réagit avec l'anhydride carbonique de l'air en dégageant de l'eau et en se retransformant en carbonate de calcium.

La carbonatation commence à partir de la surface de contact avec l'air et procède lentement vers l'intérieur;après 15 jours le rapport entre carbonate et hydrate de calcium est de 70/30. Ce rapport reste invarié pendant au moins 300 jours, et les enquêtes n'ont pas encore établi en combien de temps la carbonatation totale est réalisée.

On est tenté de penser que c'est le moment où commence la détérioration du liant parce que, aussitôt le processus de carbonatation terminé avec la transformation de tout l'hydrate en carbonate, l'eau de la pluie, chargée d'anhydride carbonique et formant de l'acide carbonique, commence par dissoudre le carbonate de calcium en produisant du bicarbonate soluble. Ce processus de dissolution ne peut pas avoir lieu tant qu'il y a encore de l'hydrate de calcium pas encore carbonisé parce qu'il neutralise l'action de l'acide carbonique.

La chaux seule est utilisée exclusivement par couches minces (badigeons), tandis que pour avoir des couches plus épaisses, à cause de son coefficient de retrait élevé pendant la phase de séchage, elle doit toujours être mélangée avec des agrégats. Chaux + agrégat + eau = mortier. Selon la réactivité des agrégats avec la chaux on obtient des mortiers séchant à l'air (normaux) ou bien des mortiers hydrauliques.

Les mortiers normaux font prise en présence d'air et leurs agrégats sont inertes, c'est-à-dire ils ne réagissent pas avec le liant, la chaux. Les agrégats inertes sont tous des agrégats organiques et, parmi les inorganiques, seulement la poudre de marbre ou de pierre et la chaux carbonatée (2). Le sable, qui réagit à long terme, ne peut pas être classé parmi les agrégats inertes, mais comme semi-inerte.

Padoue, parce qu'aussitôt mouillée, il faut la mettre en place, sinon elle s'use et brûle: raison pour laquelle elle ne fait pas prise et devient inutile (chaux hydraulique).

Suivant les sources, il devait s'agir de sables siliceux de fleuve ou de carrière, très rarement de mer, à cause de son contenu en chlorures. Par contre Palladio permet l'utilisation du sable de mer, pourvu qu'il soit ramassé derrière les dunes et lavé plusieurs fois avec de l'eau douce et en augmentant la quantité de liant à 1:2 .

La poudre de marbre avec la chaux,appelée "stuc romain" ou bien "marmorino", était largement utilisée depuis la Grèce ancienne jusqu'à nos jours. Quoique non hydraulique, ce mortier devient compact et à micropores grâce au traitement de polissage subi, et oppose par conséquent une excellente résistance aux intempéries.

En ajoutant une argile blanche (kaolin), comme elle était utilisée par les romains, elle permettait un polissage superficiel à même d'atteindre et de reproduire la splendeur du marbre. Avec ce mortier les romains préparaient les surfaces de tout genre de décoration: en relief (stucs) ou peinte. En outre, ils en couvraient les structures de murs, colonnes, chapiteaux, bases, etc. lorsqu'il n'était pas possible de se servir du marbre pour des raisons économiques.

Ce mortier a été largement employé pour obtenir des fonds blancs et lisses et y réaliser des cycles de fresques importants à toutes les époques.

A Venise aussi il était courant en tant que couche finale d'enduit en jouant deux rôles différents: bonne résistance et belle couleur marbrée.

Ce mortier était étalé en une ou plusieurs couches par ordre de granulométrie: du plus gros au plus fin. Dans les décorations extérieures des édifices ce genre d'enduit représente une imitation du marbre (comme au Teatro Argentino à Rome,etc...)

Les mortiers hydrauliques à base de chaux grasse, par contre, sont des mélanges où la nature hydraulique est déterminée par le type d'agrégat. Les agrégats hydrauliques sont des matériaux ayant subi une cuisson naturelle d'origine volcanique, tels que: les pouzzolanes incohérentes qui se trouvent en Italie centrale-méridionale, le trass en Rhénanie, la terre de Santorin sur l'île de Santorin (Théra) en Grèce, d'autres en France du Sud-Est, en Crimée, aux Açores, aux Iles Canaries, au Japon et en Indonésie. La cuisson peut être aussi réalisée artificiellement, comme pour les tessons pilés et la poudre de briques. Ces agrégats hydrauliques artificiels ont toujours remplacé les agrégats hydrauliques naturels lorsqu'ils n'étaient pas

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(2) La chaux carbonatée, utilisée en tant qu'agrégat, était employée dans l'aire grecque ancienne et byzantine.
Les sources parlent de chaux en pâte ramassée par gros tas et laissée mûrir pendant longtemps afin d'obtenir la carbonatation de la surface, en conservant de l'hydrate de calcium à l'intérieur. En mélangeant le tout on obtenait un mortier où la chaux carbonatée fonctionnait en tant qu'agrégat inerte.

disponibles sur place et les ont renforcés.

Les Romains, par exemple, utilisaient couramment des mortiers hydrauliques de pouzzolanes, et lorsqu'ils voulaient obtenir un mortier avec une propriété hydraulique plus élevée, une plus grande résistance à l'eau et une imperméabilité meilleure, comme pour revêtir des citernes, canaux, égouts, bains, aqueducs, pavements, bassins, enduits et bases externes, ils ajoutaient des tessons pilés à la pouzzolane. Ce genre de mortier, hautement hydraulique, appelé "coccio pesto", "pastellone" et "terrazzetto" à Venise, de tradition romaine, était largement utilisé pour former des enduits d'édifices placés dans des endroits avec une grande humidité de l'air, comme par exemple dans les bâtiments ruraux, surtout dans la plaine du Pô, à Venise, etc. Ces matériaux, bien qu'ils ne soient pas cimentant par leur nature, contiennent des composés de silice et alumine qui, par la combinaison avec la chaux grasse à des températures normales, forment des mortiers hydrauliques stables et très résistants à l'eau.

Selon les époques et les régions, les mortiers étaient appliqués de manière différente: en une ou plusieurs couches. Les premières couches avaient des grains plutôt grands et une quantité moindre de liant. Le rapport traditionnel avec la chaux était de 1:3 en volume. Les dernières couches avaient des grains plus petits et une plus grande quantité de liant (1:2 en volume) .

Afin d'éviter des contractions et des fêlures pendant la phase du séchage, on ajoutait normalement toujours aux enduits en pouzzolane une certaine quantité de sable dans les proportions suivantes: 2 parts de chaux, 3 parts de pouzzolane, 1 part de sable en volume.

Comme nous l'avons déjà dit, pour les mortiers traditionnels anciens on a toujours utilisé de la chaux éteinte en fosse. A présent, par contre, si l'on veut utiliser de la chaux hydratée à la place de la chaux en pâte, les proportions de mélange changent, et on aura précisément: 1 part de chaux hydratée sèche (hydrate de calcium en poudre), 6 à 7 parts d'agrégat + la quantité nécessaire d'eau (mesure en volume) .

Evidemment, dans ce cas, les enduits sont moins résistants et c'est pour cette raison qu'on y ajoute du ciment normal en formant des mortiers"bâtards", courants actuellement, mais qui ne sont pas à recommander dans les interventions de restauration à cause de leur teneur en sulphate de calcium soluble et alcali, et aussi parce qu'ils forment des barrières imperméables à la vapeur en empêchant l'échange d'humidité entre la maçonnerie et l'environnement ainsi que pour leur conductibilité thermique élevée qui est toujours négative.

Méthode d'application (Fig. 1)

Nous décrivons ici la méthode d'application traditionnelle qui est toujours courante.

a) Mouiller très bien avec de l'eau la partie de structure murale qui doit recevoir l'enduit.
b) Préparer le mortier à grains gros (de 0,08 à 2 mm) en proportion 1:3 (chaux-agrégat en termes de volume). Le mortier, assez liquide, est jeté sur le mur en exerçant de la force (crépi). Cette première couche sert à crépir et uniformiser les aspérités de la surface structurale.
c) Placer les repères: une fois l'épaisseur de l'enduit établie, on applique à hauteur d'homme un fragment de brique (environ 4x4 cm) engagé dans du mortier; ce fragment indiquera le niveau des repères suivants qui seront appliqués sur la verticale tous les 60 cm environ. Les repères verticaux sont appliqués à chaque mètre environ en les alignant au premier à l'aide du fil à plomb.
d) Les repères sont raccordés entre eux verticalement par une bande de mortier au même niveau des repères, en les nivelant à l'aide d'une tige en bois suffisamment longue. Ce sont les guides.
e) L'espace entre ces guides sera ensuite rempli de mortier toujours dans la proportion 1:3, à proportions normales d'eau. Le tout sera nivelé avec une tige en bois.
f) Cette couche (crépi) est laissée à sécher pendant un certain temps; lorsque des fêlures commencent à se produire, on mouille bien la surface et on applique la deuxième couche. Dans ce cas le mortier présente des grains plus fins (de 2 à 0,08 mm) en proportion 1:2.
g) Pour obtenir une surface d'enduit compacte et à basse perméabilité, il est nécessaire de remplir toute aspérité et porosité, même la plus petite, en étalant la dernière couche de finissage en chaux pâteuse pure à l'aide d'un outil approprié (truelle et planche à dresser).

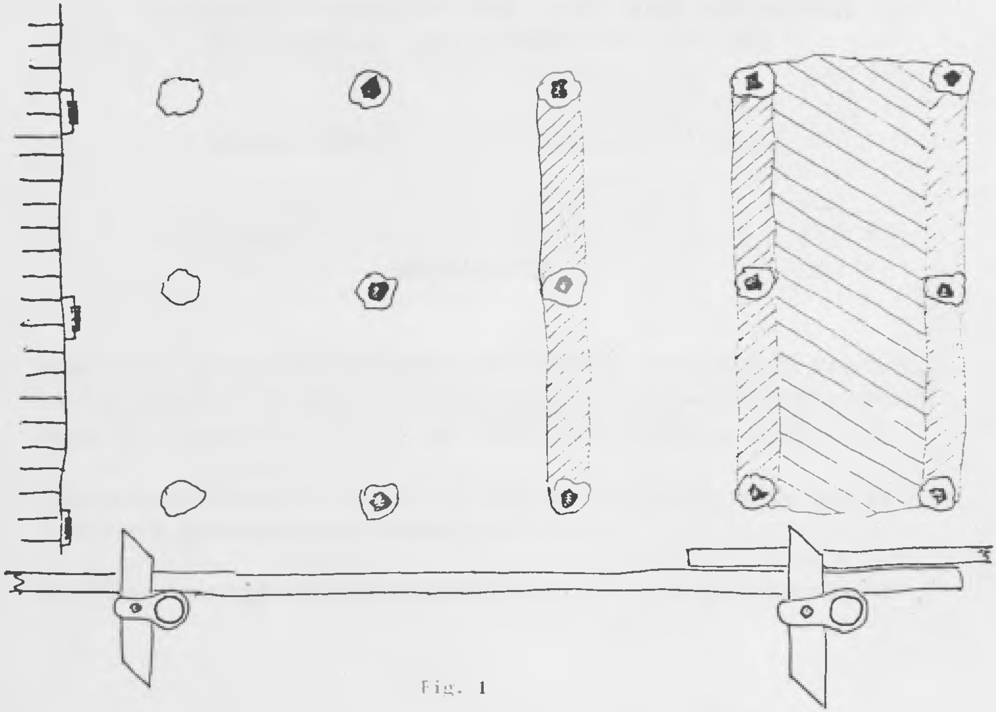


Fig. 1

A RESEARCH PROGRAMME ON THE PLASTER OF HISTORICAL BUILDINGS IN VENICE

Mario Piana[+] Emanuele Armani[++]

SUMMARY

The office of the Superintendent of historic buildings in Venice and a private fund joined forces to carry out a survey of mortars used for renderings in a large number of buildings of the city.

The survey is meant to provide data of historical value and to influence present conservation practices.

Details of sampling and documentation systems are discussed.

+ Soprintendenza ai Beni Architettonici ed Ambientali di Venezia

++ Save Venice Inc., New York

The research on plasters of Venetian historical buildings, promoted by the Soprintendenza ai Beni Architettonici e Ambientali di Venezia and financed by Save Venice, Inc., New York, started with a very precise meaning: work out a different kind of approach to the problem "plaster" as to establish a fact-based patrimony apt to fill the present deficiencies.

Such deficiencies can be identified under several aspects, either positively or negatively: approximation, empiricism, superficiality by which quite often we witness to the destruction of original historical surfaces; the scarce consideration reserved to the rôle of the plaster which represents, besides an element of noticeable aesthetique-formal importance, a determining factor to the protection of the supporting masonry, thus leading to a progressive neglect of such a technique due to the high economical expenses that frequent restorations require; the growing attention to restoration and conservation of those building elements so far neglected which requires a revaluing of the traditional materials and techniques, against the increasingly massive use of modern and not tested products and technologies.

In the "General indications to the planning directives for the lay-out of the executive project, surveys and specifications of a tender concerning restorations of historical monumental buildings" issued by the Soprintendenza and the experts of the Comune, the paragraph "plasters" states the following:

"Especially in Venice, where exterior spaces are essential to the meaning of the city itself, the preservation and remaking of plasters represents an essential operation. The good conservation of masonry, with all the processes to which it is exposed during the alternating of seasons (absorbtion, evaporation, dilation and contraction, etc.), depends also from the type of materials and the method by which a plaster is made. Too often the plastering of masonry is made with materials with a very high waterproofing degree, or stiff or cold materials with a low heat co-efficient and generally with features completely differing from those of the materials which compose both the brick and the mortar's setting bed.

Such plasters also exclude the possibility of painting with traditional materials and techniques (i.e. limewash and colours based on natural earths) and even less allow colouring on newly made surfaces nor compounding with the plaster in accordance to the traditional methods.

In the course of restoration the existing plasters, before removal, will have to be tested in their acceptability and therefore decide whether their preservation will be possible or not. In the eventuality that the plasters should be removed, fragments of ancient plasters on protected parts of façades (corbels, parts sheltered by chimneys, projections, eaves, etc.), are to be searched for to examine their consistency, quality and colour.

In general, such findings together with iconographycal proofs - whenever possible - will be used as reference for the remaking.

From these last words it is possible to see that the attempt to revalue the material "plaster" had already been started, though only at institutional level and within a well-defined context. Still missing was a methodological support and an equipment of means and instruments to improve these requirements.

The financial contribution from Save Venice, Inc., enabled the Soprintendenza to dispose of both material means and methodological support to set up the research work, establishing a communication channel between Venice and Stuttgart where the local Landesdenkmalamt for years has been coping with the vast problems concerning the preservation of plasters. The methodology set up in Stuttgart begins from the principle that every architectural-monumental building, in the course of ages, has gone through a series of modifications concerning the aesthetic material and the structural level whose identification allows to reconstruct the history of the monument: this identification establishes the fundamental premise to every restoration work. Such a principle is extended to practical research on plasters, reconstruction by mechanical removal of the various layers, the sequences of the many historical phases, and, through a contextual reading of several stratified sections, to finally trace the historical development of the monument, from its origins to our times.

Such method, however, presupposes the conservation of historical surfaces or a great deal of them: This is a condition rather rare in Venice due to the high deterioration of plasters, the acceleration of the process of decay in the course of the last century and because of the consequently more frequent use on restoration of modern materials and techniques deriving from the industrializ-ation process of the building field.

It has been therefore necessary to revise the basis of research by trying to extend to more sections the contextual reading of the stratigraphical successions, changing the German cards-index system to a more specific Venetian one on the plasters research.

Research classes, according to the various materials, have been fixed: exterior and interior plasters, stuccoes and polycromatic parts, each one with a proper technique of research, documentation and filing; a technical terminology has been set up to unify material definitions and executive normative, making them suitable to their divulging function.

Finally, the composing typologies have been identified as to classify the samples of plasters which have been removed, according to analytic parametres taking into consideration the composition, technological features, physical properties, aesthetic qualities, etc.

The outstanding features for a very efficient research are based upon three kinds of action: promptness of action on restoration worksites to avoid destruction or loss of ancient and original plaster; systematic extension, to all aspects of the problem (minor buildings, monumental architecture, etc.), of the preliminary research; incisive action to give an effective contribution of knowledge and working possibility to restoration.

Intervention on restoration sites is developed into three stages: photographic documentation, sampling, compiling of cards. The first stage allows an immediate and precise documentation on the reality of the object upon which to operate (state of the work before restoration, illustration of the stratigraphic sections, comparison after restoration). Samples represent the proof of the material's characteristics and their historical significance; the cards represent the final product on the research activity, including all necessary informations to frame the evolution of surface finishing and, therefore, also the historical development of the building.

Analysis and filing of these elements have led to quite remarkable knowledge and working contributions. Continuous attendance on restoration sites has made possible a very efficient control upon remaking of plasters and conservation of fresco traces dating back to the XV C. in the church of San Samuele, and the discovery of an ancient walled "bifora" in the Cloister of Santo Stefano. The preventive research on plasters has also provided the possibility to identify suitable materials and techniques as well as the most appropriate actions to be taken. The consideration on the results so far achieved has led to further improvement of the definition concerning analysis parametres by which proceed to the examination of every single sample.

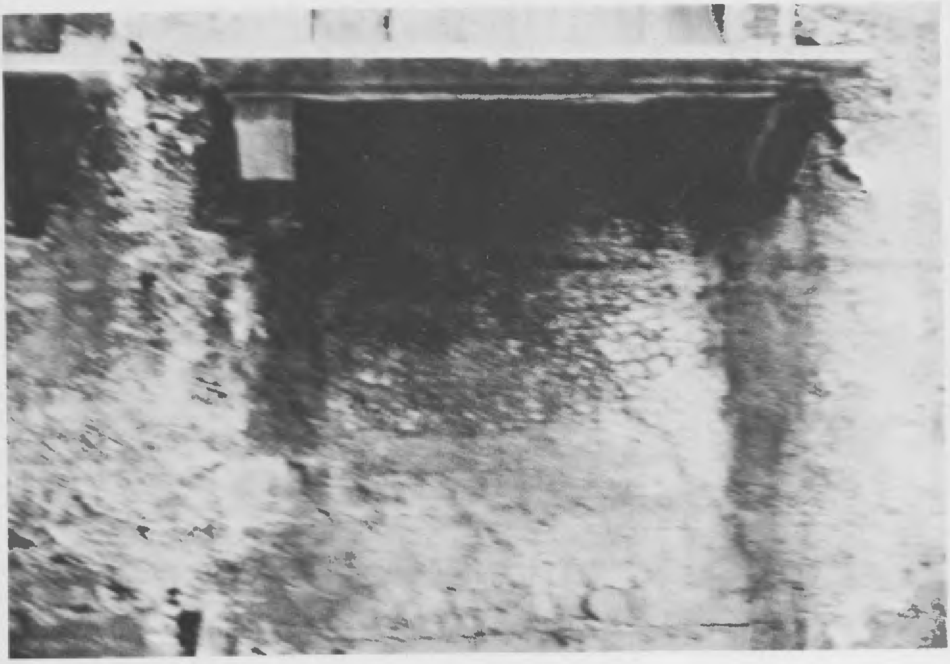
Many problems are far from being solved: quite often, especially when dealing with private restoration on minor buildings, it is almost impossible to exercise an efficient control upon the kind of work carried out, due to the fragmentation of restoration work which escapes from all attempts of research and safeguard. In other cases it is very difficult to influence decisions to be taken due to the prevailing of the economic aspect against the technical and aesthetic side of the problem, thus leading to the indiscriminate use of modern materials quite often unacceptable from the environmental point of view. Finally, the weakness of the structure for research when facing the technical-scientific part which should provide a further contribution to research and proposal.

In answer to this situation it is necessary to establish a closer coordination amongst the various institutional and research organisms, such as Soprintendenza, Comune di Venezia, Magistrato alle Acque, University, as to make public opinion and the persons in charge aware of the problem which cannot be neglected any longer, because of the many technical implications involved and the aesthetic priority it entails.

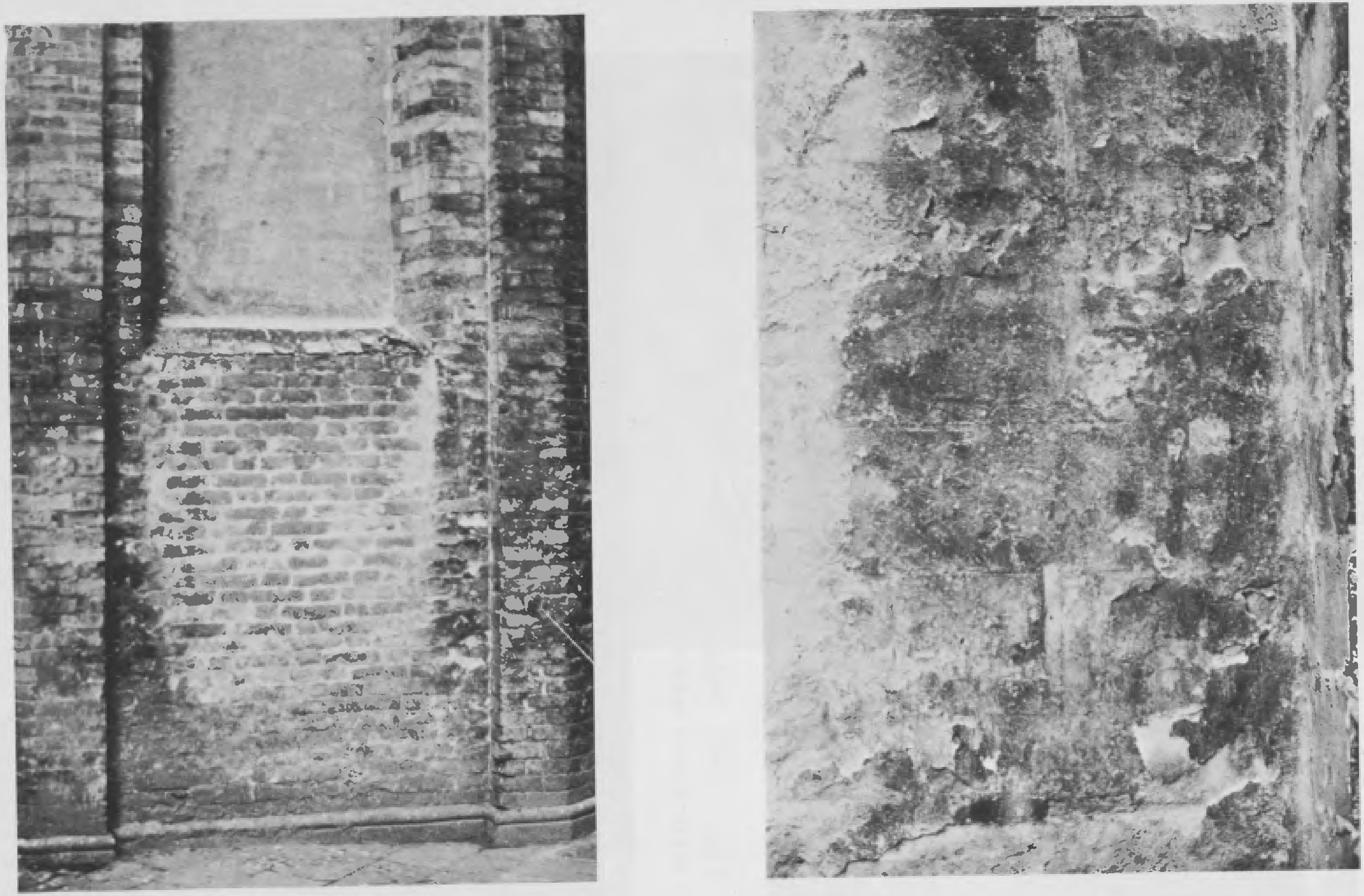
Figg. 1-2: Importance of plaster from an environmental point of view

Figs. 3-4:
Minor building with ancient decorations.

Figs. 5-6:
Minor building with
ancient decorations.



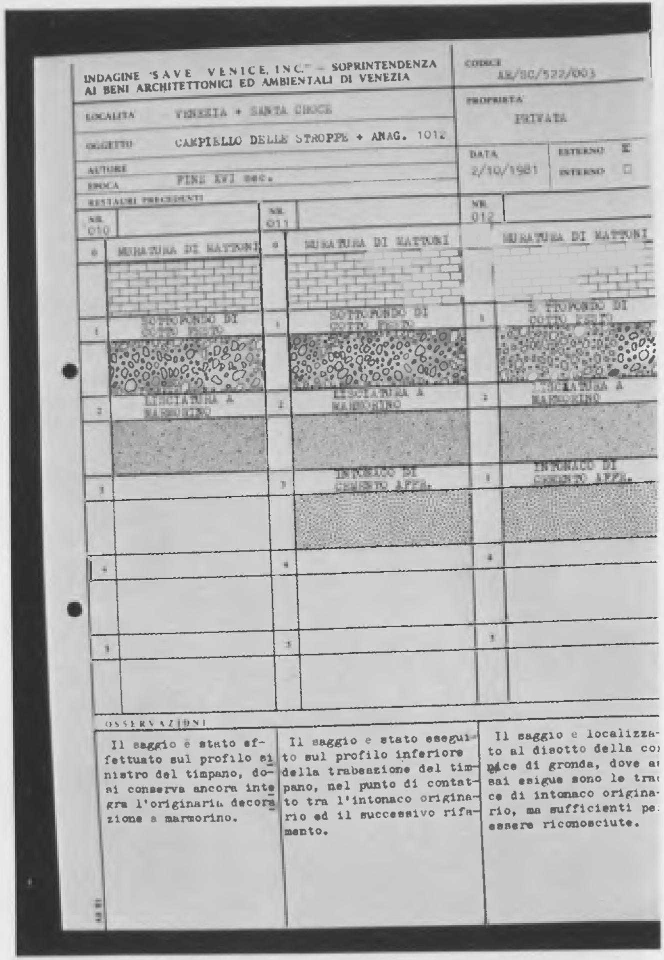
Figg. 7-8: Sample of conservation of the plaster before and after the intervention of restoration

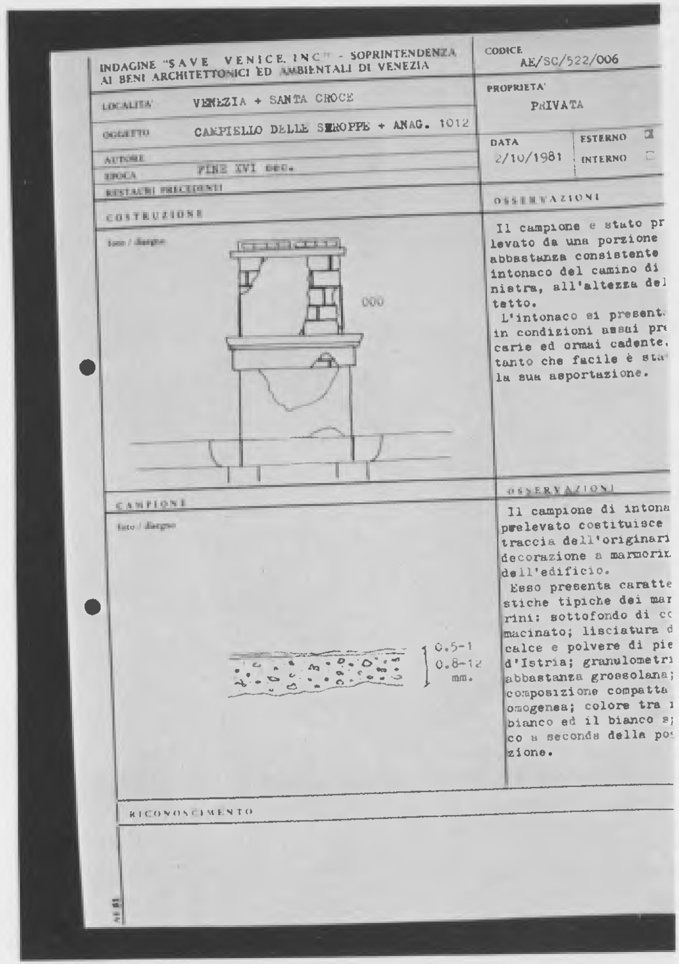


Figg. 9-10: Traces of ancient plaster
"regalzier" of Chiesa dei Carmini

Figg. 11-12: Examples of cards

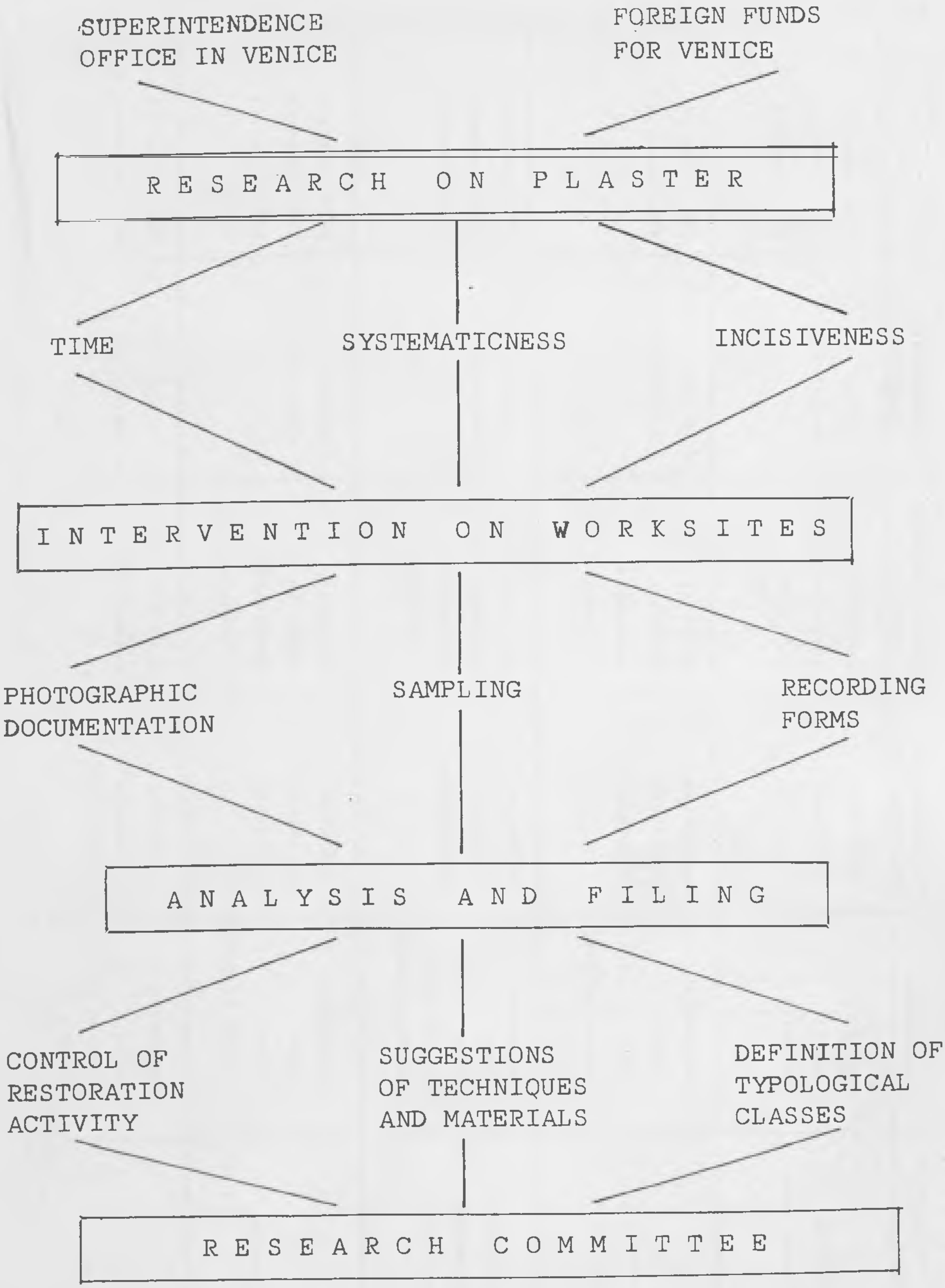
Figg. 13-14: Examples of stratigraphic sections





Figg. 15-16 : Examples of cards

Figg. 17-18: Taking and filing of sample


SUPERINTENDENCE OFFICE IN VENICE
FOREIGN FUNDS FOR VENICE
RESEARCH ON PLASTER
TIME
SYSTEMATICNESS
INCISIVENESS
INTERVENTION ON WORKSITES
PHOTOGRAPHIC DOCUMENTATION
SAMPLING
RECORDING FORMS
ANALYSIS AND FILING
CONTROL OF RESTORATION ACTIVITY
SUGGESTIONS OF TECHNIQUES AND MATERIALS
DEFINITION OF TYPOLOGICAL CLASSES
RESEARCH COMMITTEE

Parameters / Classes	Composition	Technological Characteristics	Chemical/Physical Properties	Aesthetic Qualities	Age
Pointing Setting coat	- components - oxides - binders - %	- execution - thickness - -	- porosity - permeability - adhesion -	- colour - weathering -	XIII century XIV century XV century
Fresco decoration	- components - sand - lime - earth pigment - %	- execution - thickness - finishing -	- porosity - adhesion - mechanical strength -	- colour - weathering -	XV century XVI century
"Marmorino" and smooth lime coat	- components - sand - lime - brick powder - marble powder - %	- N° layer - thickness - homogeneity -	- porosity - permeability - mechanical strength	- colour - weathering -	XVI century XVII century XVIII century
Rendering	- components - sand - lime - earth pigments - %	- execution - thickness - finishing -	- porosity - adhesion - mechanical strength -	- colour - weathering -	XVII century XVIII century XIX century
Other	- components - sand - cement - lime - %	- execution - thickness - homogeneity -	- porosity - permeability - -	- colour - weathering -	XIX century XX century

Conclusions and Recommendations

Conclusions et Recommandations

RECOMMANDATIONS

1. GROUPE DE TRAVAIL SUR L'ETUDE DES MORTIERS ANCIENS

Composé par : V. Furlan - Coordonnateur, et

E. Armani, G. Böttcher. E. Charola, M. Dupas, M. Frizot, F. Guidobaldi, G. Hyvert, H. Jedrzjewska, S. Z. Lewin, L. Mora, M. Nimmo, S. Peroni, R. Sengupta, L. B. Sickels, J. Stewart.

La discussion du groupe sur les mortiers anciens a fait apparaître que des nombreuses personnes travaillent dans le monde sur ce sujet et qu'un réel intérêt se dessine parmi des professions différentes pour ces études : architectes, restaurateurs, techniciens, chimistes, historiens, conservateurs, archéologues. Il est apparu que les besoins de connaissances en ce domaine sont très larges et très variés et que, parallèlement, les travaux effectués par les chercheurs, essentiellement dans le domaine physico-chimique n'ont pas toujours trouvé l'audience qui leur convient.

Il a semblé également à tous les participants que se faisait sentir un besoin d'échanges alors presque inexistant, entre les personnes travaillant dans cette spécialité et qu'une certaine uniformisation, ou tout au moins un consensus général devait intervenir sur un certain nombre de points soumis à discussion.

C'est ainsi que le schéma suivant a été adopté pour guider le travail ultérieur des participants, ces propositions devant promouvoir à la fois une meilleure connaissance interne du groupe des chercheurs et une communication plus satisfaisante, ainsi qu'une meilleure audience à l'extérieur, et devant accroître les possibilités réelles d'intervention et d'efficacité des recherches dans le domaine de la conservation et de la connaissance générale des monuments.

Des points de déficience actuelle dans l'étude des mortiers anciens ont fait l'objet de la discussion et les résolutions suivantes ont été adoptées :

A. Définir les buts de la recherche sur les mortiers anciens, ses motivations, ses objectifs.

Un court texte devrait montrer la diversité mais aussi l'utilité des démarches qui amènent à l'étude scientifique des mortiers anciens (historiquement de leur apparition dans la technologie à leur replacement par les ciments). L'étude des mortiers ne peut passer pour un jeu privé ou une curiosité des ingénieurs chimistes. Il s'agit de faire apparaître la justification de la recherche de même que son domaine d'application dans la conservation et la connaissance fondamentale.

B. Promouvoir la connaissance de la bibliographie, de la littérature générale, de la documentation et des archives concernant les mortiers anciens.

Ce domaine est très mal connu des chercheurs et cette spécification vise tout d'abord à éviter que ne soient poursuivies des directions de recherche déjà explorées, et aussi à faire connaître des résultat acquis mais méconnus. On veut souligner aussi par ce point l'importance de textes anciens qui répertoirent des manières, des recettes, des moyens de mise en oeuvre traditionnels qui seraient sans doute à verifier et à tester, mais qui peuvent de toute évidence servir de support à la recherche et à l'application de techniques de conservation.

C. Rédaction d'un lexique ou dictionnaire des termes usuels dans le domaine d'étude des mortiers anciens (tels que "chaux grasse","hydraulicité", "enduit", "stuc", etc..).

A l'usage de tous ceux qui sont confrontés à ces techniques sans savoir quelle est la terminologie et sa spécificité. Outre des définitions, ce lexique aborderait des caractérisations plus explicites des matériaux anciens et modernes.

D. Spécification de règles d'échantillonnage et de procédure de description ou de rapport.

Des règles minimum pour prélèvement des échantillons doivent être édictées et respectées à l'avenir, donnant toutes garanties sur la provenance du matériau et la possibilité ultérieure d'utiliser et de diffuser les résultat obtenus, permettant de les intégrer à d'autres séries, sans quoi la recherche scientifique serait inutile et inefficace. Une fiche typique facilitant la description et la documentation, comportant croquis, photographies devrait être élaborée.

E. Mise en place d'une banque d'échantillons bien caractérisés par l'analyse.

Des échantillons parfaitement connus, datés, caractérisés archéologiquement seraient réunis et proposés à l'analyse, constituant une réserve de spécimens standard. On vise ainsi à tester les méthodologies et à accroître la caractérisation d'un materiau donné. Ces échantillons devront répondre à un certain nombre de critères et de conditions imposées, tels que : abondance, homogénéité, certitude archéologique, etc...

F. Définition d'une procédure standard d'analyse.

Il s'agit de proposer des moyens d'investigation déjà testés, pour l'étude globale d'un échantillon (analyse élémentaire, analyses spécifiques comme les silicates, la calcite, etc..., examen optique, caractères physico-mécaniques, infra-rouge, microscope à balayage, etc..) ainsi que pour les études particulières requises dans le cas d'une problèmatique spéciale bien définie. Ces procédures éviteraient que des recherches inefficaces ne soient entreprises sans connaissance du travail déjà effectué sur le sujet.

Une liste de méthodes, avec ce qu'elles apportent respectivement, une méthodologie d'approche de l'échantillon doivent être proposées, justifiées par les différents buts d'investigation.

Des sujets de recherche spécifiques et systématiques actuellement insuffisamment explorés mais importants devraient être soumis à l'attention des chercherurs : rôle du tuileau, de la pouzzolane, cinétique de carbonation, rôle d'additifs mentionnés dans la littérature, etc.. avec des commentaires sur les moyens aptes à éclarer ces aspects encore obscurs.

G. Rapport d'étude et collecte des résultats.

Les connaissances acquises sont restées jusqu'à maintenant assez confidentielles et insuffisamment diffusées. Il conviendrait d'adopter des mesures pour la publication rapide et très accessible de tels résultats ayant une incidence sur les questions du ressort de l'ICCROM (restauration et conservation). Il manque actuellement une publication du type Newsletter disposée à accueillir des commentaires méthodologiques spécialisés dans notre domaine. Sa création pourrait être envisagée dans le cadre du groupe.

H. Propositions pour la conservation et la restauration.

L'apport de l'étude des mortiers anciens dans le champ de la connaissance des monuments doit être souligné et des propositions nouvelles devraient être formulées pour l'utilisation de méthodes, de techniques, de matériaux, de recettes qui apparaîtraient, à la lumière des études ci-dessus, plus aptes à satisfaire les objectifs et les exigences premières de l'intervention sur les monuments anciens.

Les responsables de laboratoires présents se proposent de collaborer à la mise en oeuvre des points cités. La formation d'un comité ICCROM des mortiers anciens est adoptée et Monsieur S. Z. Lewin est proposé pour la coordination du groupe.

2. GROUPE DE TRAVAIL SUR L'ETUDE DES MORTIERS ET COULIS POUR LA CONSERVATION DES MACONNERIES ANCIENNES

Composé par : A. Bouineau - Coordonnateur, et

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Ce groupe de travail s'est borné à définir un plan général des recherches considérées nécessaires pour la rationalisation de l'emploi de mortiers et coulis dans la conservation des structures anciennes.

On recommande à l'ICCROM et aux autres organisations internationales compétentes d'encourager toute activité de recherche de base et appliquée dans ce domaine, tant au niveau national qu'international, en particulier en créant des systèmes de liasons et d'échanges d'information entre les laboratoires scientifiques et technologiques, les architectes intéressés à la conservation, les sociétés commerciales qui assurent l'éxécution de ces travaux de conservation et les fabricants de liants, coulis et mortiers.

A. Domaine de l'étude. Types de mortiers et coulis.

I. Mortiers

1. Mortier pour enduit.
2. Mortier de rejointement.
3. Mortier de protection horizontale ("capping").
4. Mortier provisoire à capillarité élevée (pour l'extraction de sels des maçonneries.
5. Mortier de ragréage.

II. Coulis

1. Renforcement des maçonneries;
   a) renforcement de la structure
   b) renforcement des composants.
2. Injection derrière un parement (peinture murale, etc...)
3. Injection pour assurer l'étanchéité.
4. Remplissage des vides.

B. Caractérisation des mortiers et coulis. Propriétés souhaitées et risques à éviter.

Le matériau doit être compatible avec la structure originale et son environnement. En particulier les points suivants doivent être considérés :

a. Résistance mécanique.
b. Apport de matériaux dangereux.
c. Comportement vis-à-vis de l'eau (liquide et vapeur).
d. Dilatation thermique ou due à l'eau.
e. Modifications dues au vieillissement.
f. Mise en oeuvre (devrait être aussi simple et fiable que possible).
g. Limites de la réversibilité (le degré de réversibilité qu'on peut attendre d'un mortier ou coulis employé en conservation devrait être étudié).
h. Facteurs esthétiques (enduits, mortiers de ragréage).
i. Marquage des matériaux ajoutés dans la restauration (dans les matériaux mêmes ou par documentation).

Cette liste doit être considérée incomplète et purement indicative.

C. Mesure des caractéristiques et critères de sélection.

I. Mesures

1. Examen in situ du batîment avant, pendant et après traitement.
2. Essais de laboratoire. Standardisation.

II. Critères de sélection

L'élaboration de critères de sélection des matériaux sur la base des caractéristiques requises par les divers emplois de mortiers et coulis est la tâche finale, la plus difficile de la recherche.

Elle peut être accomplie seulement après qu'une expérience commune de travail de plusieurs laboratoires employant des méthodes d'essais standardisées ait montré la corrélation entre la performance sur le terrain et les essais de laboratoire.

Il est vrai, cependant, qu'une élaboration provisoire de critères de sélection, basée sur les connaissances déjà acquises, est souhaitable à court terme pour influencer au plus tôt l'attitude des fabricants de liants, des architectes et des sociétés commerciales travaillant dans la conservation des bâtiment anciens.

RECOMMENDATIONS ^^

1. WORKING GROUP FOR THE STUDY OF ANCIENT MORTARS

Group members : V. Furlan - Coordinator and

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The discussion of the group on ancient mortars brought out that many people are working on this subject throughout the world and that real interest in this field is appearing among professionals of different disciplines : architects, restorers, technicians, chemists, historians, curators and archaeologists. It also seemed that the need for knowledge in this area is extensive and diverse, while the work done by researchers has not yet been accorded proper attention. It appeared to all the symposium participants that there is a need for exchange (almost non-existent to date) between people working in this particular branch of conservation, and that a certain standardization, or at least a general consensus on several points is required.

The following draft was adopted as a guideline for the participants' further work. These suggestions should promote both better information and communication within the research groups and a better audience outside. Real possibilities of intervention and effectiveness through research should also be increased in the field of conservation and in the general knowledge of historic buildings.

Present weak points in the study of ancient mortars were discussed by the working group, and the following recommendations were adopted :

A. Definition of the aims of ancient mortar research, reasons and objectives.

A short paper should describe the variety, as well as the importance of activities stimulating scientific study of ancient mortars

^^ Translation from the French Original

(historically, from their advent in building technology to their replacement by cements). The analysis of mortars cannot be considered simply as a private game or a curious enthusiasm of chemical engineers. It is important to justify the reasons for this research, showing how it applies to conservation and contributes to fundamental knowledge.

B. Promotion of familiarity with bibliography, general literature, documentation and archives concerning ancient mortars.

Researchers are often poorly acquainted with this field so this point is meant primarily to avoid duplication of research work and also to make known results that have been achieved but not recognized. The importance of ancient texts should also be stressed here, as they provide information on traditional methods, materials, receipts, and working techniques. This information must undoubtedly be verified and tested, but it can be a useful support in research and application of conservation techniques.

C. Drafting a glossary, or dictionary, of common terms for the study of ancient mortars (e.g. "rich lime", "hydraulicity", "plaster", "stucco", etc.).

This would be intended for use by those who are interested in these techniques but not acquainted with the terminology and its specific usage. In addition to definitions, this glossary will deal with the most obvious characteristics of ancient and modern materials.

D. Specifications for sampling and procedural rules for description and reporting.

Minimum rules for the taking of samples, providing full guarantees of the material's origin, should be agreed upon and then respected in the future. The possibility of eventual use and distribution of any results obtained should also be considered, so they can be included in other studies. Otherwise, scientific research will be useless and ineffective. A standard form to facilitate description and documentation, including photographs and sketches, should be developed.

E. Foundation of a bank of samples that have been thoroughly analysed.

Samples of known provenance, dated and with an archaeological pedigree, should be collected and analysed, so as to build up a reserve of standard specimens. Another aim here is to test our methodology and expand the means of characterizing a given material. The samples should meet a certain number of criteria and conditions, such as quantity, homogeneity, definite archaeological data, etc.

F. Definition of a standard analytical procedure.

Tested methods of investigation should be proposed for the complete analysis of samples (basic analysis, specific analyses such as those for silicates, calcite, etc..., optical examination, mechanical and physical characteristics, infra-red, electronic scanning microscope, etc.). Particular studies, required by special, well defined problems, should also be included. These procedures will avoid useless duplication of effort on the same subject. A list of methods, with their respective implications, and a methodology of sample handling should be proposed and justified according to the aims of the investigations. Specific, systematic research topics, currently unexplored but of importance, should be brought to the attention of researchers, together with comments on methods that could clarify these still obscure aspects. Such topics might be the role of brick powder or pozzolanic ash, the kinetics of carbonation, the role of additives mentioned in the literature, and so forth.

G. Study reports and collection of results.

Knowledge acquired to date has been treated as fairly confidential and inadequately distributed. It would be advisable to adopt measures for prompt and readily available publication of such results, which might influence ICCROM's further activities (restoration and conservation). What is currently missing is a Newsletter type publication, where specialized technical commentary in our field could be collected. Creation of such a newsletter might be a working group activity.

H. Proposals for conservation and restoration.

It should be stressed that the study of ancient mortars contributes to the field of knowledge of historic buildings, and new propositions must be formulated for the utilization of methods, techniques, materials, and receipts that appear, in the light of the above studies, most likely to satisfy the objectives and primary exigencies of interventions on historic buildings. The laboratory heads here present intend to cooperate in implementing the points mentioned. It was decided to form an ICCROM Committee on Ancient Mortars, and Mr. S. Z. Lewin was proposed as committee coordinator.

2. WORKING GROUP FOR THE STUDY OF MORTARS AND GROUTS FOR THE CONSERVATION OF ANCIENT MASONRY

Group members : A. Bouineau - Coordinator and

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This working group limited its task to the definition of a general plan of researches which are considered necessary for the rationalization of the use of mortars and grouts in the conservation of ancient structures.

ICCROM and other international organizations are recommended to encourage every activity of basic and applied research in this field, at both the national and international level, in particular creating liaison systems and promoting exchange of information among scientific and technical laboratories, architects involved in conservation, commercial firms that carry out conservation work, and the manufacturers of binders, grouts and mortars.

A. Field of study. Types of mortars and grouts.

I. Mortars

1. Mortar for rendering.
2. Mortar for pointing.
3. Mortar for capping.
4. Temporary mortar with high capillarity (for extraction of salts from masonry).
5. Mortars for fillings and stuccoes.

II. Grouts

1. Reinforcement of masonry;
   a) reinforcement of the structure
   b) reinforcement of the constituents.
2. Injections behind a facing (mural paintings, etc.).
3. Damp-proofing injections.
4. Filling of voids.

B. Characterization of mortars and grouts : desirable properties, risks to avoid.

The material should be compatible with the original structure and its environment. The following points should be considered in detail :

a. Mechanical resistance.
b. Formation of dangerous by-products.
c. Behaviour with respect to water (both liquid and vapour).
d. Expansion due to heat or water.
e. Modifications due to weathering.
f. Application (which should be as simple and reliable as possible).
g. Limits of reversibility (the degree of reversibility that can be expected of a mortar or grout applied in conservation should be studied).
h. Aesthetic factors (renderings, fillings and stuccoes).
i. Marking of materials added during conservation work (in the materials themselves or by documentation).

This list must be considered incomplete and purely indicative.

C. Measurement of properties and selection criteria.

I. Measurement

1. Field survey of the building before, during and after treatment.
2. Laboratory tests. Standardization.

II. Selection criteria

The development of selection criteria for materials is the final and most difficult task of such a research. Criteria must be based on the properties required in the various context in which mortars and grouts are employed.

This task can be accomplished only when the correlation between field performance and laboratory tests has been established through the common work experience of several laboratories using standardized methods of analysis.

Nevertheless, it is true that a provisional formulation of selection criteria, based on knowledge already acquired, is desirable at short term, in order to have an immediate influence on manufacturers of binders, architects and commercial firms working in the conservation of historic buildings.

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Mortars - cements - grouts . . .

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